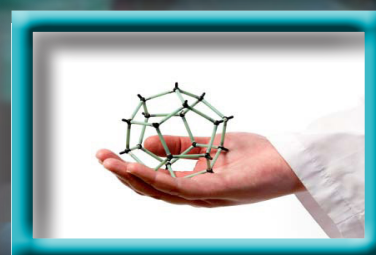
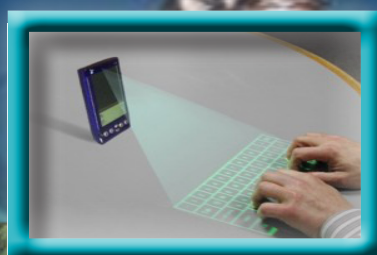


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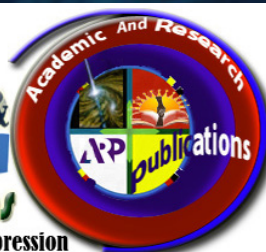
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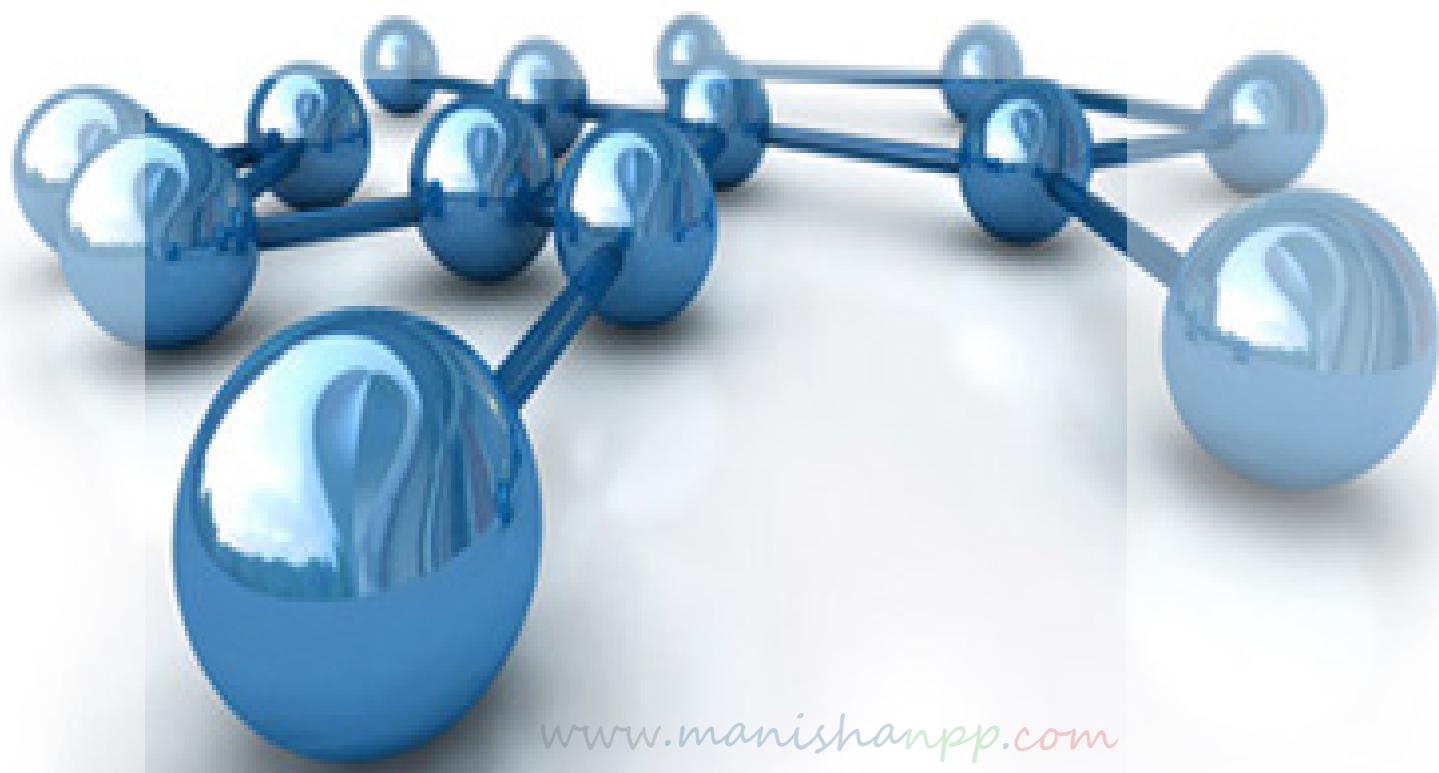
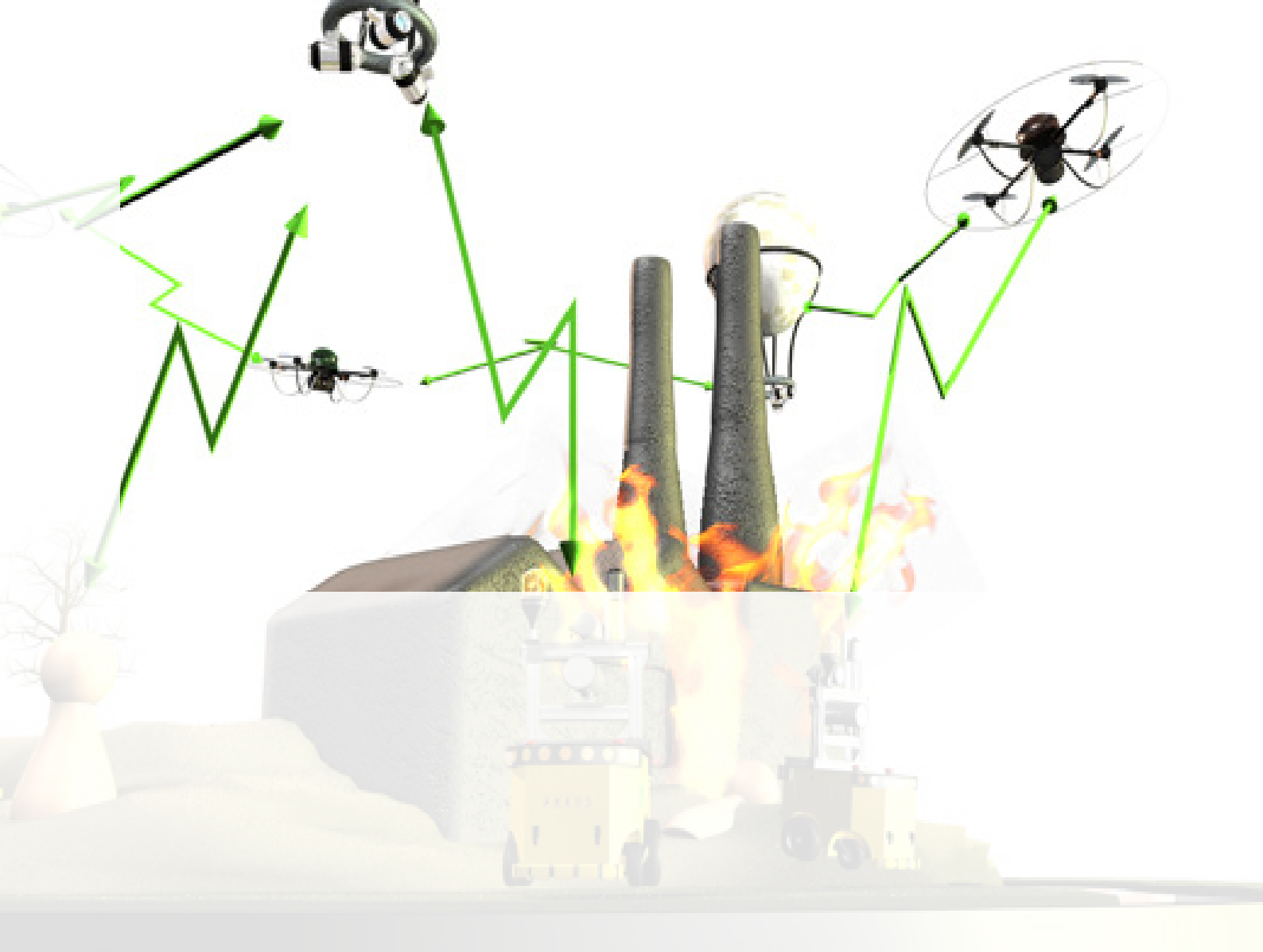
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# SYNTHESIS AND CHARACTERIZATION OF MAGNETIC COBALT NANOPARTICLES USING PLURONIC P123 AS SURFACTANT AT ROOM TEMPERATURE

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## ABSTRACT

The aim of the present work is to synthesize cobalt nanoparticles (CoNPs) at room temperature using pluronic P-123 as surfactant. This method is fast and simple which can be done under room temperature. Compared with other methods it is cost effective and easy to prepare. The synthesis of cobalt nanoparticles by reduction of cobalt salt with sodium borohydride as a reducing agent with the combination of ethanol and stabilizing agent P123. Characterization of cobalt nanoparticles were carried out by XRD, SEM and EDX. The size of the nanoparticles is predominantly within the range of 50-100 nm.

**Key Words :** Synthesis of Co nanoparticles, Synthesize Cobalt Nanoparticles (CoNPs), P123 (Pluronic acid) , Magnetic Nanoparticles.

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## INTRODUCTION

The properties of nanoparticles are extremely important because as the size of the particle decreases the properties are significantly altered from those of their bulk <sup>[1]</sup>. Magnetic nanoparticles, especially Co, Fe, Ni and their alloys, have attracted increasing interest among researchers due to their promising applications in high-density magnetic recording media <sup>[2]</sup>. Among these magnetic metal nanoparticles, the cobalt nano-

particles exhibit single-domain magnetism, high-saturation magnetization, and high coercive force; therefore, these have great potential applications in the field of magnetic record and memory <sup>[3]</sup>. The crystal phase of nanoparticles is a key factor for application in magnetic recording, The Co nanoparticles has a richer crystal phase diagram with three nearly isoenergetic crystal structures: face-centered cubic (FCC), hexagonally close-packed (HCP), and epsilon <sup>[2]</sup>. Jey-



adevan *et. al.*, reported that cobalt nanoparticles have the advantage of lower annealing temperatures (300-500 °C) with high magnetism [4]. The magnetic properties of NP's are extremely dependent on sample type, magnetization direction and crystallinity of the samples [5]. The key factor for the magnetic property is the presence and absence of magnetic domain. The magnetic properties of nanoparticles are determined by many factors: **(a)** the chemical composition, **(b)** the type and the degree of defectiveness of the crystal lattice, **(c)** the particle size and shape, **(d)** the morphology, **(e)** the interaction of the particle with the surrounding matrix and the neighboring particles [6]. Recently, these CoNPs have been synthesized by a variety of methods including thermal decomposition, gas vapor condensation, and reduction of cobalt salt. Generally, in most types of nanoparticles are prepared by these method (sol-gel processing, hot spraying, evaporation condensation, matrix isolation, laser-induced vapor phase reactions and aerosols) control of size and size distribution is not possible [7]. For most of these strategies, however, complicated procedures, sophisticated equipment and rigorous experimental conditions are required. Thus, further development of the synthetic techniques into practical routes to large quantities of nanostructures from a diversified range of materials, rapidly, and at reasonably low costs, still requires great inventiveness. In contrast, unconventional methods based on chemical synthesis might provide an alternative and interesting approach for generating nanostructures in terms of material diversity, cost, and the potential for high-volume production [8]. Magnetic metal-

lic nanoparticles can be synthesized from metal salts using strong reducing agents, namely, alkali metal dispersions in ethers or hydrocarbons, alkali metal complexes with organic electron acceptors (e.g., naphthalene), sodium borohydride ( $\text{NaBH}_4$ ) and other complex hydrides. By using  $\text{NaBH}_4$  in aqueous solutions at room temperature, both homo- (Fe, Co, Ni) and heterometallic (FeCo, FeCu, CoCu) nanoparticles were obtained as amorphous powders containing substantial amounts of boron [9]. Shao *et. al.*, reported synthesis of CoNPs using nontoxic cobalt acetate tetrahydrate as a precursor material [10]. Shin reported  $\text{NaBH}_4$  reduction technique for CoNPs synthesis using cobalt chloride as precursor in aqueous solution [11].

In the present project work, an easy and inexpensive chemical synthesis route has been proposed. The synthesis of CoNPs by reduction of cobalt salt with  $\text{NaBH}_4$  as the reducing reagent with the combination of ethanol and surfactants (P123) has been carried out at room temperature. The samples are characterized by X-ray Diffraction (XRD) and SEM measurements. The elemental analysis and chemical composition are studied by Energy Dispersive X-ray Spectroscopy (EDX).

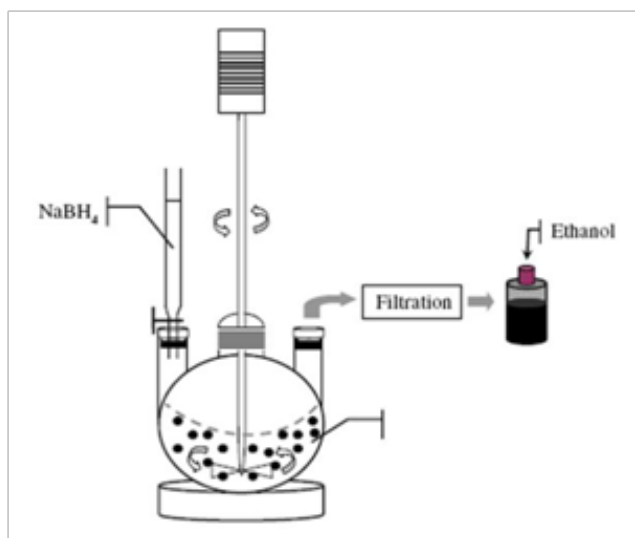
## Experimental Work and Methodology

## MATERIALS AND METHODS

Cobalt acetate, P123 (Pluronic acid) and ethanol were obtained from Aldrich chemicals. Milli-Q water was used throughout the work.



## Synthesis of Co nanoparticles



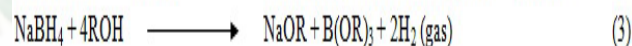
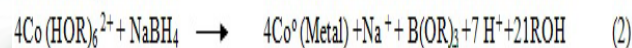
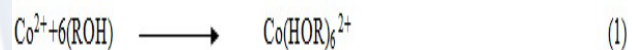
**Figure 1 : Experimental Setup for the synthesis of CoNPs.**

CoNPs were prepared through chemical reduction of cobalt acetate  $\text{Co}(\text{CH}_3\text{COO})_2$  in ethanol. To prevent the product from aggregation, a surfactant has to be added at the beginning of each synthesis. In a typical experimental design, 3.7gms of cobalt acetate was dissolved in ethanol. Pluronic acid (P123) was dissolved in ethanol. The above prepared solutions were added in a three-necked round bottom flask fitted with a mechanical stirrer at the rate of 500 rpm under constant stirring as shown in the above diagram (Fig. 1). On stirring a pink colour emulsion was obtained. Then  $\text{NaBH}_4$  solution was added dropwise to the above solutions at room temperature under stirring at 500 rpm for 30 min. The solution turned to black colour which showed the reduction of cobalt acetate. The black colour solution was allowed to settle down at room temperature. The black precipi-

tate of the CoNPs was separated by centrifugation at 5000 rpm for 10 min. The remaining solution was decanted, and the precipitate was rinsed with ethanol twice followed by centrifugation in the same rpm. The supernatant was decanted and the CoNPs were isolated in wet form. The processes of filtration and pulverization were the significant and controlling steps. The black precipitate was filtered through 0.22 micron filter paper using suction pump and the wet product was taken out in watch glass for the process of pulverization and drying done at cooled condition to avoid ignition of the particles. Finally, air stable CoNPs powder was obtained at room temperature. The CoNPs synthesized by this method were very stable for a long time.

## Reaction Mechanism

The reduction of transition metal ions by  $\text{NaBH}_4$  is a ubiquitous reaction useful for the production of ultrafine particles of metals and metal borides. However,  $\text{NaBH}_4$  reduction chemistry is complex and the nature of the products depends strongly on the reaction conditions used [12-13].



When the cobalt salt was dissolved in ethanol,  $\text{Co}(\text{HOR})_6^{2+}$  was formed with a pink colour (Reaction 1). Reaction 2 shows that the replacement of  $\text{Co}(\text{HOR})_6^{2+}$  by  $\text{NaBH}_4$  and the colour of the solution turned to black with the evolution of  $\text{H}_2$  gas.

## RESULTS AND DISCUSSION

### Characterization of CoNPs

CoNPs was characterized by the techniques such as XRD, SEM and EDX. Figure 2 shows the typical XRD pattern of CoNPs synthesized. The XRD pattern shows broad peaks which indicate that our sample is amorphous in nature. To

narrow down the peaks annealing of sample is done at 800 °C for 4 hr. The XRD pattern shows broader peaks due to the outer covering of organic surfactants on the surfaces on CoNPs even after heating at 800 °C. It clearly indicates that the sample is amorphous due to its hard covering which can prevent the oxidation of metal in air and avoid aggregation.

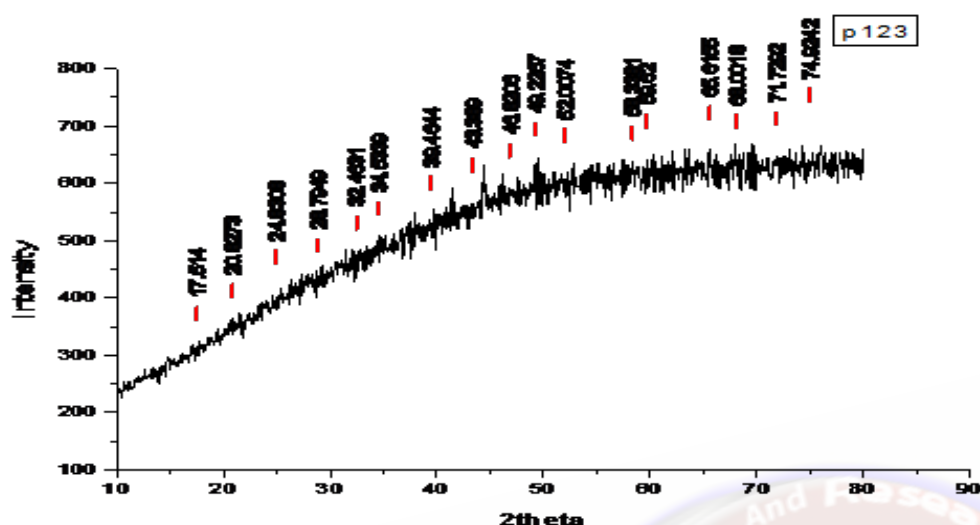
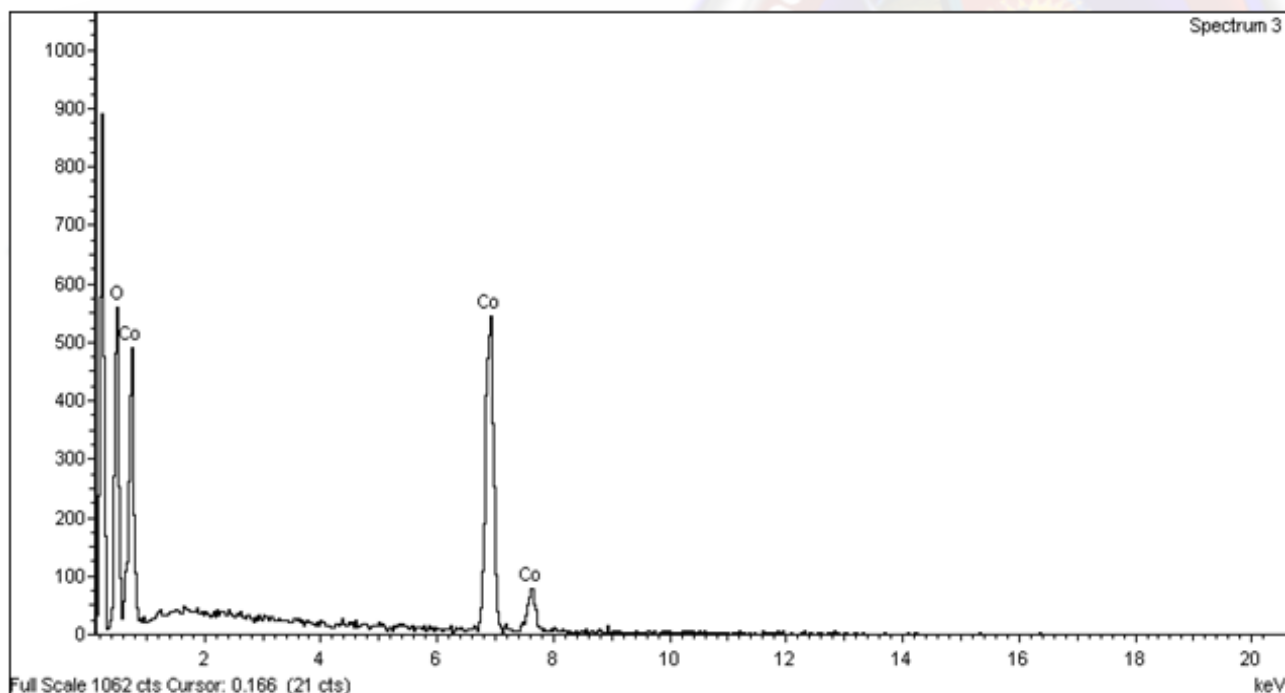


Figure 2: XRD diffractogram of CoNPs

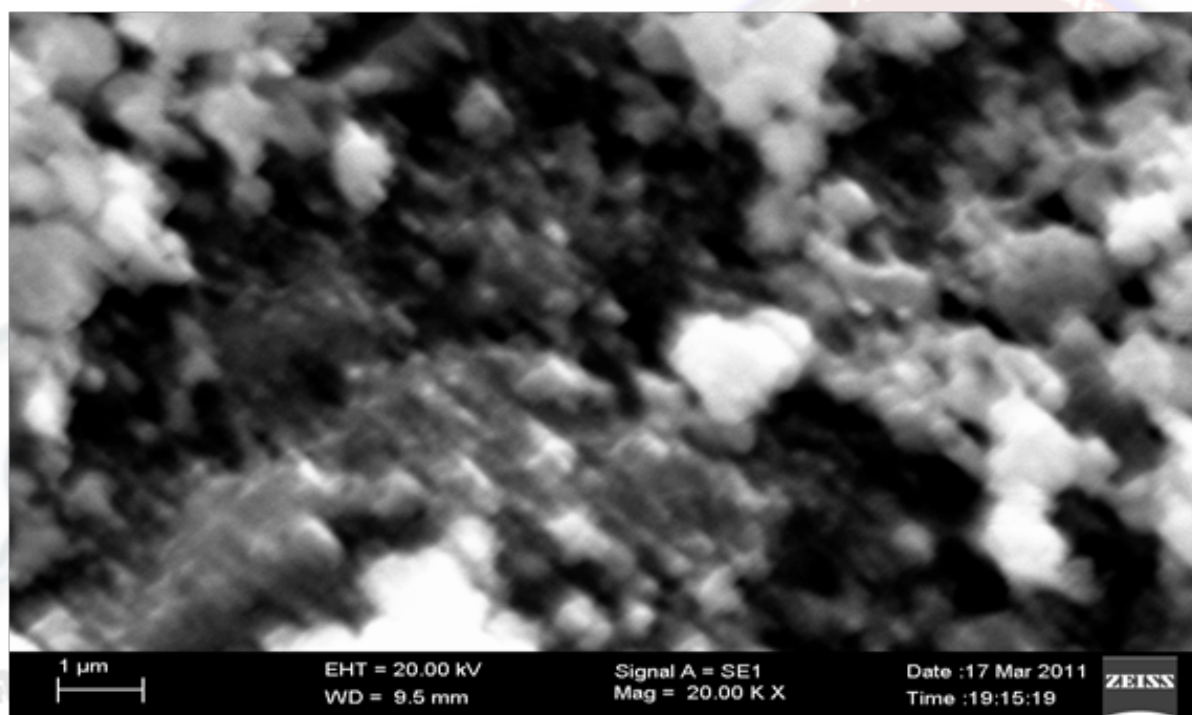


Element	Weight %	Atomic %
O K	13.58	36.67
Co K	86.42	63.33
Totals	100.00	

**Figure 3: Energy Dispersive X-ray (EDX) microgram of CoNPs**

The EDX spectra of the CoNPs shows that it consists of 86.42% of cobalt and 13.58% of oxygen (weight %) while 63.33% of cobalt and 36.67% of oxygen (Atomic %). The typical SEM image of the synthesized CoNPs (Fig. 4) shows

size within less than 100 nm. Due to magnetic dipole-dipole interactions of the individual particles more aggregation of the nanoparticles causes the large surface area and bigger sized particles.



**Figure 4: SEM microgram of cobalt nanoparticles.**



## CONCLUSION

The CoNPs prepared by borohydride reduction method using surfactants are very stable at room temperature for long time. In the borohydride reduction method for the synthesis of nanoparticles ethanol is used as the solvent and P123, are used as a capping agent. P123 is a good chelating agent and also a good stabilizer. P123 is a protective copolymer which stabilizes the CoNPs by preventing their aggregation and affords protective coatings by preventing their re-oxidation. Finally, the particles obtained were stable and black in color. XRD results showed that the cobalt nanoparticles synthesized was amorphous in nature. The EDX spectra of the CoNPs showed that it consisted of 86.42% cobalt and SEM images showed its particle size less than 100 nm. Hence P123 may be a smart choice to prepare stable and smaller sized particles of magnetic CONPs at room temperature.

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# COLORIMETRIC DETECTION OF $\text{Hg}^{+2}$ USING DIASTASE CAPPED SILVER NANOPARTICLES

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## ABSTRACT

Diastase capped silver nanoparticles (AgNPs) were prepared via a green reduction route and used as colorimetric sensors for the detection of  $\text{Hg}^{+2}$ . The synthesized AgNPs were characterized by using UV-visible, XRD and TEM analysis. The obtained AgNPs showed selective colorimetric detection of potentially toxic  $\text{Hg}^{2+}$  ions in aqueous media at micromolar level. The proposed colorimetric method i.e. sensing system has effectively been used for the detection of  $\text{Hg}^{+2}$  in tap water samples.

**Key Words :** Silver Nanoparticles, Colorimetric Sensors, Mercury, Diastase.

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## INTRODUCTION

Now-a-days, metal nanoparticles such as gold and silver have received more attention among all the nanomaterials because of their optical, electronic and magnetic properties. Due to these extraordinary properties, silver nanoparticles (AgNPs) has applications in optoelectronics <sup>(1)</sup>, pharmaceuticals <sup>(2)</sup>, catalysis <sup>(3,4)</sup>, electronics <sup>(5)</sup>, antimicrobial products <sup>(6,7)</sup>, sensing <sup>(8)</sup>, therapeutics <sup>(9-11)</sup>, and photonics <sup>(12)</sup>. Recently, consumer products containing AgNPs are commonly available in the market because of their antimicrobial activity towards large range of fungi, bacteria and low cost of production. For instance, Ag-

NPs are being used in coatings, clothes, food, bandages and containers as disinfectants and deodorizers <sup>(13,14)</sup> as well as an insecticide for pest control <sup>(15)</sup>.

On the other hand, the selective colorimetric detection of hazardous heavy metal ions present in the environment is of growing significance. For instance, divalent mercury ion ( $\text{Hg}^{+2}$ ) is one of the most common toxic heavy metals existing in the nature and causes health threats affecting central nervous system, brain and kidney <sup>(16,17)</sup>. According to the United States Environmental Protection Agency (USEPA) the recommended levels of  $\text{Hg}^{+2}$  in water is 2 ppb, whereas the World Health Organization



(WHO) and Bureau of Indian Standards (BIS) suggested it as 1 ppb. Hence, it is important to detect and determine the  $\text{Hg}^{2+}$  levels in the biological and environment samples with very good selectivity and sensitivity.

Traditional methods used for the direct quantitative detection of mercury includes the gas chromatography, cold vapor atomic fluorescence spectrometry and absorption spectroscopy<sup>(18,19)</sup>. But, these methods are time consuming and expensive instruments are needed with complex sample preparation methods. Therefore, it is greatly advantageous to develop simple and rapid methods for measuring  $\text{Hg}^{2+}$  levels in the environment. However, several reports have been published for the immediate detection of  $\text{Hg}^{2+}$  using chromophores<sup>(20)</sup>, noble metal NPs<sup>(21-23)</sup>, fluorophores<sup>(24)</sup>, polymer<sup>(25)</sup> to evade the complex sample preparation and instrumentation.

Herein, we describe the use of diastase, a thiol group containing enzyme as reducing and stabilizing agent for the synthesis of AgNPs in alkaline pH. The prepared AgNPs are stable and acted as selective colorimetric sensor for the detection of  $\text{Hg}^{2+}$  in aqueous media.

## Experimental Section

### MATERIALS AND METHODS

Silver nitrate, sodium hydroxide, metal salts and all other organic solvents were purchased from sigma aldrich chemicals, Bangalore. Metal stock solutions were prepared by dissolving metal salts in double distilled water and were further used whenever necessary. Diastase was pur-

chased from CDH (P) Ltd., Delhi, India.

### Preparation of Silver Nanoparticles

Diastase capped AgNPs were prepared as per our reported procedure elsewhere<sup>(26)</sup>. In brief, 1 mL diastase solution (1%) of pH 12 was added to 5 mL of 1 mM  $\text{AgNO}_3$  solution and stirred at room temperature for about 10 min using a magnetic stirrer. Formation of AgNPs was known by the change of colour from colourless to reddish brown and was monitored by using UV-visible spectroscopy.

### General Procedure for the Colorimetric Determination of $\text{Hg}^{2+}$

To examine the colorimetric detection of  $\text{Hg}^{2+}$ , the as-synthesized AgNPs were diluted three times with double distilled water. Investigation of ability of diastase stabilized AgNPs for detection of  $\text{Hg}^{2+}$  was carried out by the addition of 200  $\mu\text{L}$  of 1 mM metal ion solutions to the 300  $\mu\text{L}$  of three times diluted AgNPs solution. The changes in absorption were monitored at room temperature using UV-visible spectrophotometer and all the readings were taken after 5 minutes of metal ions addition to AgNPs solution.

### Preparation of Simulated Water Samples

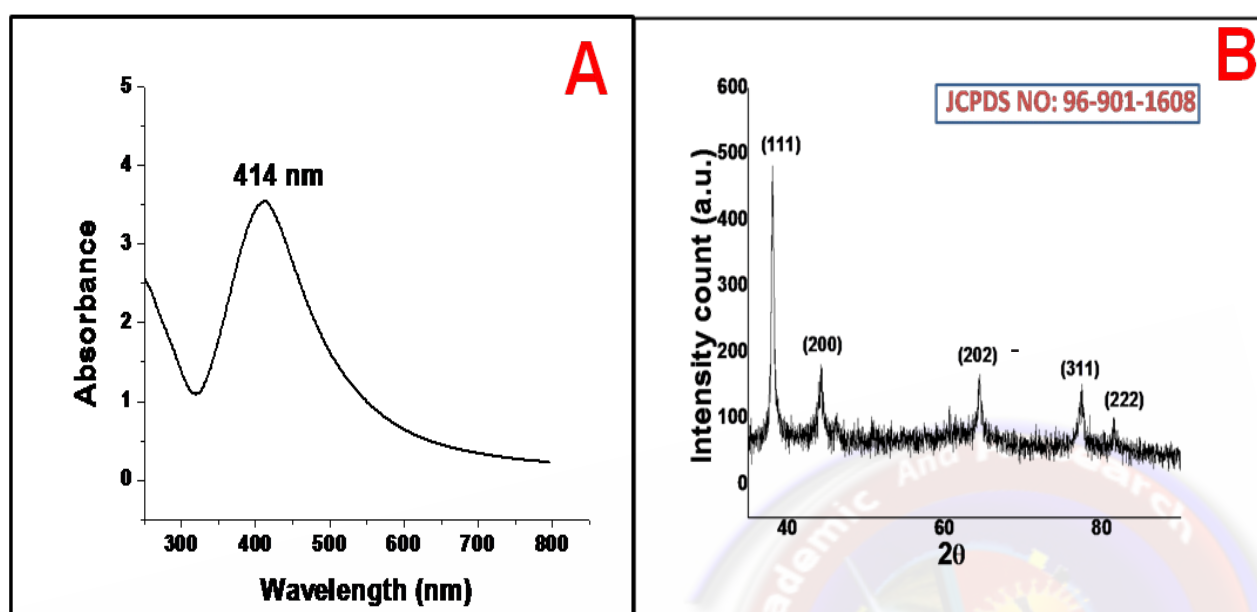
Tap water samples were collected and filtered through a cellulose nitrate filter paper and a suitable volume of  $\text{Hg}^{2+}$  solutions was spiked to make simulated real samples. The analysis of spiked water samples was carried out by the proposed procedure using UV-visible spectrophotometer.

## RESULTS AND DISCUSSION

Diastase, an enzyme containing thiol groups have used for the synthesis of AgNPs, played the key role as both reduc-

ing and stabilizing agent. When diastase solution was added to the  $\text{AgNO}_3$  under alkaline conditions, the colour of the solution changed from colorless to yellow and then to reddish brown due to the surface plasmon resonance of the AgNPs. From the UV-visible spectra of as-synthesized AgNPs, the presence

of an intense SPR absorption peak at 414 nm indicated the change of  $\text{Ag}^+$  to  $\text{Ag}^0$  (Fig.1A). The free thiol groups existing on the diastase surface might be responsible for the reduction of  $\text{AgNO}_3$  to AgNPs and the formed NPs were stabilized by the carboxylic groups of diastase.

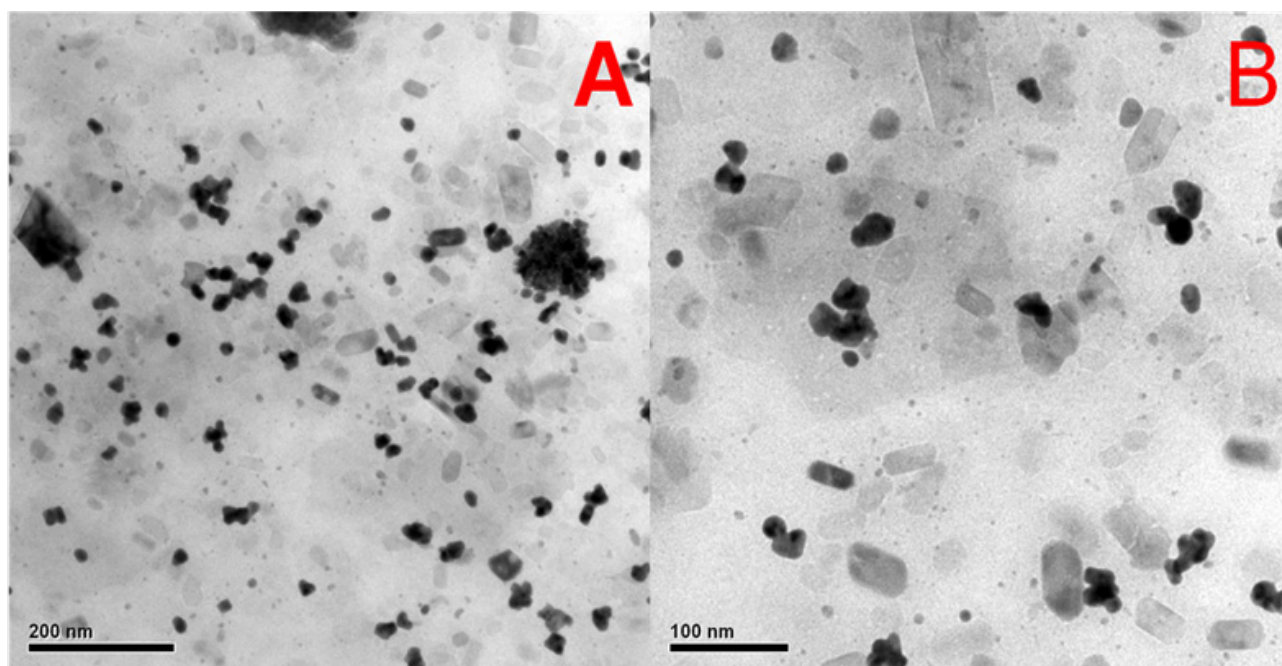


**Figure 1: (A) UV-visible spectra (B) XRD pattern of AgNPs synthesized with diastase**

The purified AgNPs were characterized by using powder XRD. Figure 1B shows the XRD pattern of the diastase stabilized AgNPs. The formed AgNPs showed different diffraction peaks at  $2\theta$  values of 38.1, 44.2, 64.39, 77.35 and 81.37 which corresponds to the characteristic planes (111), (200), (220), (311) and (222) respectively, indicating the face centered cubic crystal structure of

AgNPs (JCPDS NO: 96-901-1608). The morphology of the prepared NPs was studied under TEM. Figure 2 shows the TEM images of the AgNPs prepared with the diastase. TEM images represent the presence of anisotropic spherical and rod shaped nanoparticles. It also shows the formation of rod shaped nanoparticles by the aggregation of spherical smaller nanoparticles.





**Figure 2: TEM images of the diastase capped AgNPs**

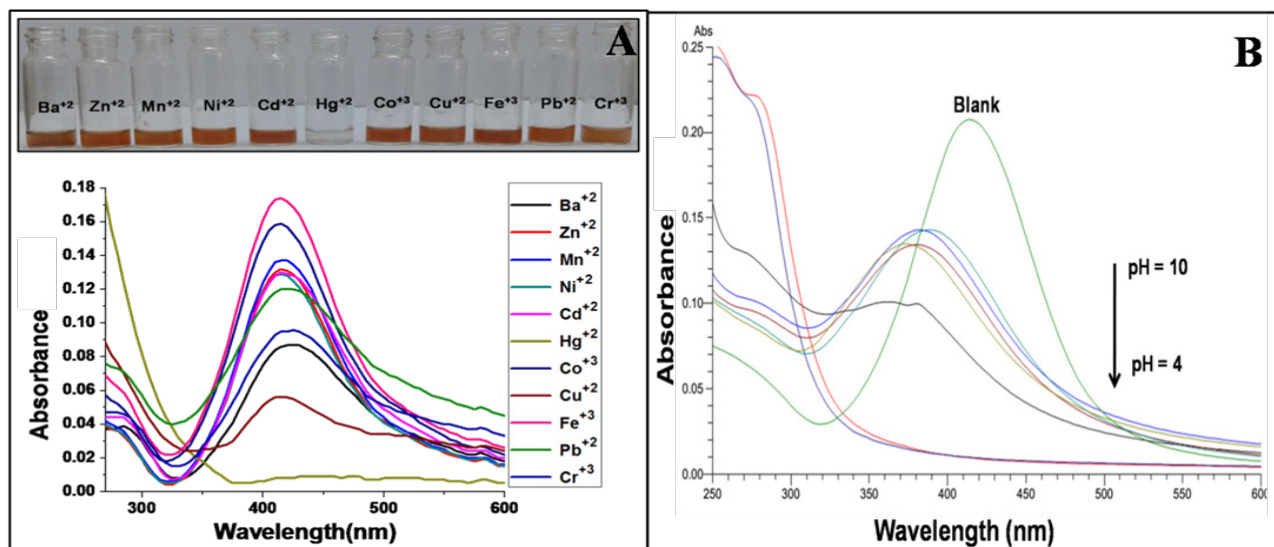
### Colorimetric Determination of $\text{Hg}^{+2}$

In order to study the selectivity of AgNPs towards other metal ions, we examined the color change with different metal ions such as  $\text{Cu}^{+2}$ ,  $\text{Pb}^{+2}$ ,  $\text{Mn}^{+2}$ ,  $\text{Co}^{+2}$ ,  $\text{Cd}^{+2}$ ,  $\text{Ni}^{+2}$ ,  $\text{Fe}^{+2}$ ,  $\text{Ba}^{+2}$ ,  $\text{Zn}^{+2}$ ,  $\text{Cr}^{+2}$  at 1mM concentration each. The AgNPs solution existed as reddish brown with the addition of 1mM each of these metal ions except  $\text{Hg}^{+2}$  ions. After addition of 1mM of  $\text{Hg}^{+2}$  ions it resulted in the disappearance of the color of AgNPs solution immediately. UV-visible absorption spectra showed the disappearance of peak at 414 nm (Figure 3A), which may be due to the binding of  $\text{Hg}^{+2}$  ions onto the surface of AgNPs to move diastase enzyme aside.

Further, the colorimetric responses of the

AgNPs system towards  $\text{Hg}^{2+}$  were studied at different pH of 4, 5, 6, 7, 8, 9 and 10. A noticeable color disappearance was observed for AgNPs at pH '4' and '5' with the addition of  $\text{Hg}^{2+}$  (80  $\mu\text{M}$ ). Addition of  $\text{Hg}^{2+}$  (80  $\mu\text{M}$ ) to AgNPs at pH '4' and '5' resulted in the large decrease in the absorbance, compared to that at higher pH (6, 7, 8, 9, 10) indicating that pH '4' and '5' are the optimum condition for AgNPs sensing system for detection of  $\text{Hg}^{+2}$  (Figure 3B). This may be attributed to the fact that in the basic solutions the presence of hydroxyl groups may interact with the metal ions and results in the decreased interaction between mercury ions and AgNPs surface. Hence, pH '4' and '5' are the most favorable conditions for the present sensing assay.

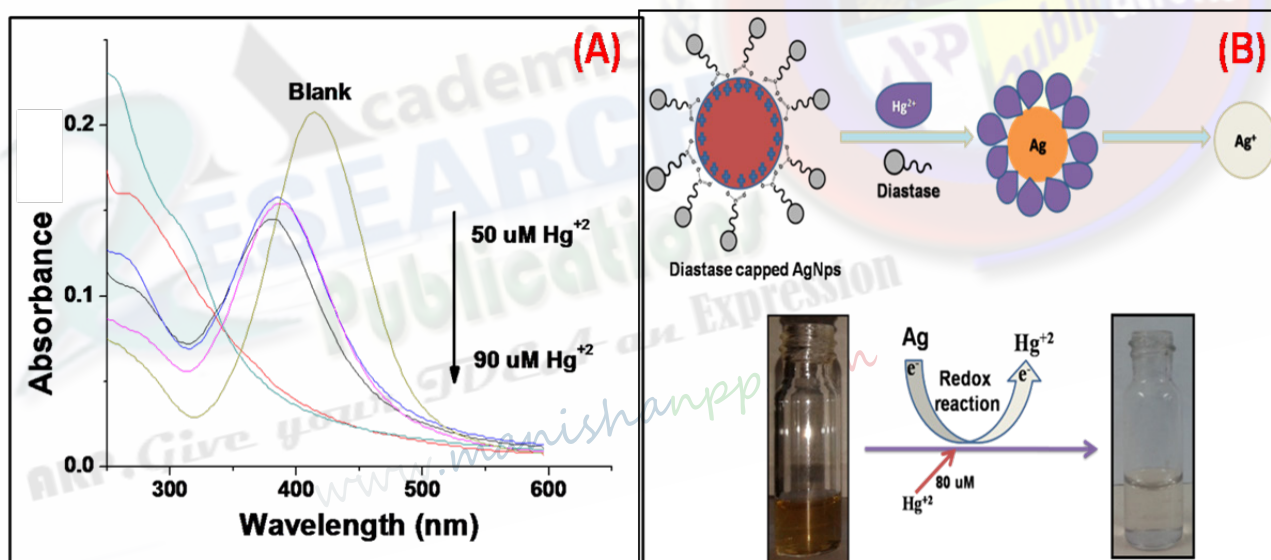




**Figure 3: (A) UV-visible spectra showing the colorimetric response of AgNPs towards various cations (B) Response of AgNPs system upon addition of  $\text{Hg}^{2+}$  ions ( $80 \mu\text{M}$ ) at various pH**

Further, different concentrations of  $\text{Hg}^{2+}$  ions were added to AgNPs solution, in order to assess the limit of detectable concentration of  $\text{Hg}^{2+}$  in aqueous solutions. UV-visible absorption spectra of AgNPs and the related color change were recorded after the addition of different concentrations of  $\text{Hg}^{2+}$  (Figure 4A). The distinctive SPR peak of AgNPs was used to determine the limit of detection for  $\text{Hg}^{2+}$  by using UV-visible

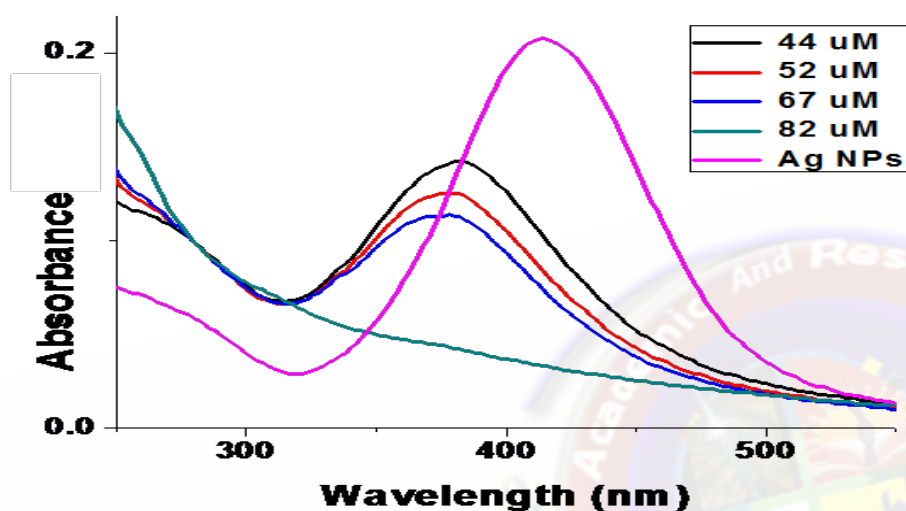
spectroscopy. From Figure 4A, it is confirmed that the concentration of  $\text{Hg}^{2+} < 80 \mu\text{M}$  did not show complete color change of AgNPs. The absorbance at  $414 \text{ nm}$  decreased with increasing concentration of  $\text{Hg}^{2+}$  added, suggested more  $\text{Hg}^{2+}$  ions binding to AgNPs surface <sup>(27)</sup>. These results conclude that the lowest concentration of  $\text{Hg}^{2+}$  that can be detected by AgNPs colorimetrically was  $80 \mu\text{M}$ .



**Figure 4: (A) UV-vis absorption response of AgNPs system upon addition of  $\text{Hg}^{2+}$  ions ( $50\text{--}90 \mu\text{M}$ ) (B) Possible mechanism of  $\text{Hg}^{2+}$  detection by AgNPs.**

Synthesized AgNPs were characterized after and before the addition of  $\text{Hg}^{+2}$  ions using UV-visible spectroscopy. Typical SPR absorption peak of Ag nanocolloids after 2 times dilution was found at 414 nm and the solution was yellowish in color without the presence of  $\text{Hg}^{+2}$ , but became colorless with the addition of 80  $\mu\text{M}$  of  $\text{Hg}^{+2}$ . Based on the UV-visible data, the sensing mechanism of AgNPs may be based on the redox reaction between AgNPs and  $\text{Hg}^{+2}$ . The standard reduction potential of  $\text{Hg}^{+2}$  (0.85 V) is higher than the  $\text{Ag}^+$  (0.8 V) and hence the added  $\text{Hg}^{+2}$  will be bound on the surface

of AgNPs to move the diastase molecules away from the surface of AgNPs, resulting the redox reaction between  $\text{Hg}^{+2}$  and silver. Mercury (II) ions may be reduced radiolytically in aqueous AgNPs solution and a mercury layer will be formed around AgNPs. Figure 3A also showed the peak broadening and blue shift of the SPR band, when 80  $\mu\text{M}$  of  $\text{Hg}^{+2}$  was added to the AgNPs dispersion, which might be the result of adsorbed metallic mercury atoms formed by the reaction of  $\text{Hg}^{+2}$  with AgNPs <sup>(28)</sup>. The proposed mechanism of  $\text{Hg}^{+2}$  detection by AgNPs system is shown in Figure 4B.



**Figure 5: UV-vis response of AgNPs system towards  $\text{Hg}^{+2}$  in tap water matrix**

Further, the diastase stabilized AgNPs system was evaluated for the determination of  $\text{Hg}^{2+}$  in tap water matrix. The amount of  $\text{Hg}^{+2}$  in the environmental samples such as tap water is very less and hence a potential practical assay is necessary. These tap water samples were free of  $\text{Hg}^{+2}$  or the amount may be very less for detection. Hence, the samples were spiked with various concentrations of mercury within the range of 1-100  $\mu\text{M}$  and the atomic absorption

spectroscopic data were taken to know the concentrations of unknown solutions which was found to be 44, 52, 67, 82  $\mu\text{M}$  (Figure 6, Table 1). Figure 5 shows the ability of AgNPs sensing system for the detection of  $\text{Hg}^{+2}$  in the tap water. The limit of detection of AgNPs based sensing system for the detection of  $\text{Hg}^{+2}$  ions in tap water samples is 80  $\mu\text{M}$ . This result further suggested the great potential of present method for the sensing of  $\text{Hg}^{+2}$  in tap water samples.

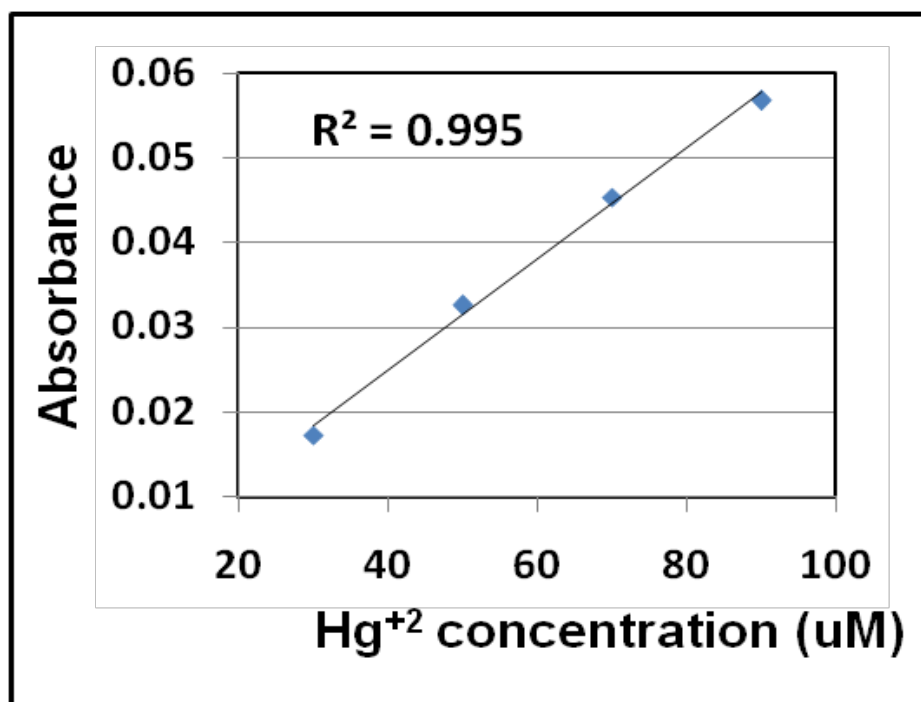


Figure 6: Calibration plot of standard Hg<sup>2+</sup> solution (30, 50, 70, 90 µM) obtained by atomic absorption spectroscopy

Table 1 AAS absorbance values of simulated Hg<sup>2+</sup> samples prepared in tap water matrix

Sample	Absorbance	Molarity (µM)	Recovery of spiked Hg (II) in tap water (%)
Unknown 1	0.0276	44	110
Unknown 2	0.0329	52	104
Unknown 3	0.044	67	111.6
Unknown 4	0.053	82	102.5



## CONCLUSION

In this paper we have showed the efficiency of diastase capped AgNPs for the colorimetric detection of  $\text{Hg}^{+2}$ . This AgNPs system showed high selectivity for  $\text{Hg}^{+2}$  ions over other alkaline, alkali and transition metal ions with a limit of detection of  $80 \mu\text{M L}^{-1}$ . The high stability and easy synthesis of the protein directed synthesized AgNPs allocate this method to be very simple to apply. This method offers low cost and simple way of  $\text{Hg}^{+2}$  detection in the environmental samples without any further modifications.

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# SIZE CONTROLLED SYNTHESIS OF SILVER NANOPARTICLES - a review

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## ABSTRACT

Size controlled synthesis of metal nanoparticles remains a narrow part of research in the field of nanotechnology even after the development of various methods for their synthesis. The intrinsic properties of the nanoparticles can be controlled by tuning their size and shape, which has generated great potential applications in several fields of engineering and science. This review mainly emphasises on different methods reported so far for the production of silver nanoparticles (AgNPs) with tunable sizes. This review also discusses about the various parameters responsible for tuning the size of AgNPs.

**Key Words :** Size Controlled Synthesis, Silver NPs, Tuning of Size.

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## INTRODUCTION

Recently, the development of techniques for the preparation of new class of nanoscale materials is becoming greatest importance in nanotechnology. This is mainly because of the day by day raising in the significant role of nanomaterials in various fields of science such as electronics, chemistry, biology and physics. The unique extraordinary mechanical, thermal electronic, optical and biological properties made them unusual from other bulk scale materials. But still the synthesis of nanomaterials with desired features is the greatest challenge in the field of nanoscience.

Generally nanomaterials are featured

by (i) Quantum confinement effects (ii) large number of particles per unit weight, and (iii) high surface to volume ratio. All these features of nanomaterials result in extremely diverse properties from those of bulk materials. For example, the properties of materials that change from bulk to nanometer range are optical property (1), magnetic properties (2) and solubility (3), melting temperature (4). For example, gold nanoparticles (AuNPs) shows ruby red or blue color depending on their size whereas the conventional gold is yellow (5). It is well known that the large surface area per unit weight is a significant feature of nanomaterials.

In general, methods for synthesis of na-



nomaterials can be largely classified into two different categories: (i) top down <sup>(6,7)</sup> and (ii) bottom up <sup>(8,9)</sup> approaches. Generally, top down approach includes the physical synthesis methods such as laser etching <sup>(10)</sup>, lithographic techniques <sup>(6,7)</sup> and ball milling <sup>(11)</sup>, where the materials in bulk state are scaled down until the nanoscale or desired size is obtained. But, the problems such as hardness with ball milling techniques and high cost involvement of lithographic techniques in designing very small nanomaterials made them less suitable for industrial applications <sup>(6,7,11)</sup>. In contrast, bottom up approach commonly includes the biological and chemical synthetic methods. Here, the nanomaterials of required shape and size can be synthesized from their bulk precursors using a biological or chemical reduction reaction. During the course of reduction, the building blocks of the nanomaterials will be formed first which later will be assembled to form the final nanostructure. The chemical and physical parameters can be changed in order to control the nucleation and growth rate, and also the addition of external capping agent is done to stop the growth process in attaining the nanomaterials of required shape and size <sup>(12)</sup>. Additionally, the nanoparticles (NPs) of small size can be separated from large size by using centrifugation <sup>(13)</sup> in solution phase synthesis.

Nanoparticles are the nanoscale materials that exist in the size range between 1-100 nm. During the Roman period, metal nanoparticles were used as decorants in glasses, where glasses showed different colors when they were viewed through reflected and transmitted light due to the presence of NPs <sup>(14)</sup>.

Noble metal NPs have been commonly

used in different applications such as surface enhanced Raman scattering (SERS) <sup>(15)</sup> and Plasmonics <sup>(16,17,18)</sup>. Additionally, they have been used in various bio-applications such as antimicrobial <sup>(19)</sup>, immunological labeling <sup>(20,21)</sup> and X-ray contrast agents <sup>(22)</sup>. On the other hand, Palladium (Pd) and Platinum (Pt) NPs have been established as very good catalysts for different hydrogenation reactions. Hence, the growing use of metal NPs in different areas of science is becoming a promising area of attention. Metal NPs (MNPs) of different sizes can be fabricated using various biological and chemical methods. Moreover, MNPs exhibits different properties with change in their size. For example, the phenomenon of Surface plasmon resonance (SPR) exhibited by noble MNPs varies with the size of gold and silver NPs. Hence there is a raising demand for the synthesis of MNPs of desired size and shape.

Ahmad *et. al.*, have prepared the AgNPs of different size by ultrasonication method using k-carrageenan as reducing agent. They have found that the irradiation time played the key role in tuning the NPs size <sup>(23)</sup>. In the same way, Bonatt *et. al.*, have developed a temperature dependent method for tuning the size and shape of the AgNPs using the fruit seed extract of cashew-apple. They have proposed that the size of NPs increased with increase in the temperature of the reaction system <sup>(24)</sup>. Zong *et. al.*, have proposed a kinetically controlled seed mediated synthesis method for size tuning of AgNPs using glucose as reducing agent. The average sizes of the obtained NPs were found between 20-200 nm <sup>(25)</sup>.

Panacek *et. al.*, have reported the synthesis of size controlled AgNPs within the





diameter range from 28 to 77 nm using D-maltose as reducing and stabilizing agent. The comparative catalytic activity of the prepared AgNPs against the methylene blue degradation showed the high efficiency of small size NPs over larger ones <sup>(26)</sup>. Similarly, Suganya *et. al.*, have developed a *Spirulina platensis* extract mediated biosynthetic method for the synthesis of AgNPs of different sizes. Authors have shown the comparative antimicrobial activity of synthesized NPs and proved the improved activity of AgNPs with decrease in size. They also reported that the activity was size and dose dependent <sup>(27)</sup>.

Alternatively, reaction conditions such as pH also played an important role in tuning the size of NPs. For example, Parameshwaran *et. al.*, have varied the pH of the reaction mixture containing beta vulgaris peel extract and AgNO<sub>3</sub> to obtain the NPs of different sizes <sup>(28)</sup>. Jeong *et. al.*, have synthesized AgNPs of two different sizes (10 nm, 100 nm) using sodium citrate as reducing agent and their cytotoxic and antimicrobial properties were studied. Change in the concentration of sodium citrate used for the reduction played key role in the preparation of nanoparticles of required size. The comparative cytotoxicity and antimicrobial activity studied against the *Methylobacterium* bacteria suggested the size and dose dependant activity of NPs. Authors suggested that the size and amount of the AgNPs were important parameters to be considered before using in the commercial products such as blood-contact medical devices <sup>(29)</sup>.

Chen *et. al.*, prepared a highly dispersive AgNPs of different sizes by chemical method where PVP was used as reduc-

ing agent and particles size was tuned by varying the solvent (water, ethanol and ethylene glycol) used to carry out the reaction. Introduction of organic solvent such as ethanol decreased the size of the NPs formed which may be due to the decrease in the collision between the AgNPs. It was also found that the smaller NPs were obtained with ethylene glycol which because of the longer chain length of ethylene glycol compared to ethanol <sup>(30)</sup>. Goswami *et. al.*, have prepared AgNPs of different sizes via a chemical method by using NaBH<sub>4</sub> as reducing agent in the presence of PVP and sodium citrate. Morphology of the NPs was controlled by varying the amount of the stabilizer PVP during synthesis process. They have found that the smaller NPs showed the good electrochemical response towards the cholesterol sensing <sup>(31)</sup>.

Zahran *et. al.*, have proposed a new method to prepare AgNPs of different sizes using sodium alginate. They have tuned the size of the nanoparticles by varying the temperature of reaction system (60-80 °C). This may be due to the higher temperature played an key role in accelerating the redox reaction between silver ions and sodium alginate which finally led to the change in the size of the NPs <sup>(32)</sup>.

Xu *et. al.*, have prepared flower-like Ag mesostructures with the size range of 200 nm to 700 nm via a simple seed-mediated approach. This size-tunable synthesis was easily attained by adjusting the concentration of Ag seeds. The formation of Ag mesostructures was attained by the assembly of primary Ag nanosheets, which exhibited a flower-like





architecture. Compare to the previous approach for flower-like Ag mesostructures, the present method was simple and reliable using green procedure and size controllability. These flower-like Ag mesostructures revealed large surface area and was served as highly efficient catalysis for the  $\text{NaBH}_4$  reduction of p-nitrophenol to p-aminophenol <sup>(33)</sup>. Lin *et. al.*, have synthesized highly fluorescent AgNPs with various sizes by the microemulsion method. The size of the AgNPs was controlled easily by altering the molar ratio of water to surfactant in microemulsion. The molar ratio and reaction time could influence the maximum absorbance wavelength and the absorbance of AgNPs, as well as the fluorescence emission intensity <sup>(34)</sup>.

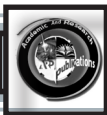
Reithofer *et. al.*, have synthesized a size controlled AgNPs in UV irradiation method with using ultra short peptides such as Ac-LIVAGK $\text{NH}_2$  <sup>(35)</sup>. Tang *et. al.*, have synthesized AgNPs in polyol process where PVP was used as a capping and PEG as a reducing agent. Ratio of the PVP to PEG played an important role in tuning the size of the AgNPs. Higher the PVP to PEG ratio bigger size NPs formation was observed <sup>(36)</sup>. Oh *et. al.*, have synthesized AgNPs of controlled size and shape in seed-mediated growth method by using ascorbic acid and citrate. The size of the nanostructures was varied by changing the concentration of citrate and ascorbic acid. Larger size NPs was obtained when the concentration of ascorbic acid and citrate ions was higher. Catalytic studied have shown that the large surface area of the bumpy nanostructures would be highly attractive as efficient heterogeneous catalyst <sup>(37)</sup>.

Piella *et. al.*, have shown that the varia-

tion in the concentration of tannic acid in the reaction medium resulted in the AgNPs of different sizes. The average sizes of the obtained NPs varied from  $10.1 \pm 0.9$  nm to  $46.1 \pm 8.3$  nm. The catalytic studies showed the high catalytic efficiency of smaller NPs <sup>(38)</sup>. Kang *et. al.*, have developed a PVA mediated synthesis of size controlled AgNPs by electron irradiation method. In this method particle size was tuned by varying the energy of electron beam and current. Particle size was increased with increase in the dose of electron beam and current. They have also studied the increase in the sizes of the NPs by increase in the concentration of PVA <sup>(39)</sup>.

Similarly, Zhang *et. al.*, have synthesized silver nanocrystals with uniform sizes in droplet micro reactors through seed-mediated growth using glycolaldehyde as a reducing agent. The average diameter of the obtained NPs was 30-100 nm <sup>(40)</sup>. Zao *et. al.*, have used sodium borohydride solution for the synthesis of AgNPs of different sizes in chemical route and found that with increasing the volume of silver seed used in reaction the obtained size of the NPs was decreased <sup>(41)</sup>. Jacob *et. al.*, have synthesized AgNPs of different sizes by chemical method using water-ethylene glycol mixture. They have varied the concentration of ethylene and water to tune the size of NPs. Increasing the water-ethylene glycol ratios the formation of silver atom cluster decreased, resulting the decrease in the size of NPs <sup>(42)</sup>.

Meneses *et. al.*, have prepared a size and shape controlled synthesis of AgNPs via a chemical method using the secondary amines. They have identified that the addition of 1 or 5 equivalents of ethylenedi-



amine as additional capping agent notably decreased the average size of the particles <sup>(43)</sup>. Cassar *et. al.*, synthesized size tunable monodispersed AgNPs by green method using plant seeds. In this method different quantities of seed extract were added to the AgNO<sub>3</sub> solution for tuning the size <sup>(44)</sup>. Mikhlin *et. al.*, have used the different molar ratios of AgNO<sub>3</sub> and NaBH<sub>4</sub> for tuning the size of NPs <sup>(45)</sup>.

Liu *et. al.*, have developed Cacumen Platycladi extract mediated synthesis of AgNPs of different sizes in micro reactor method. Size of the NPs increased with increase in the extract quantity which might be due to the increased availability of plant biomolecules for capping <sup>(46)</sup>. Mashayekh *et. al.*, have prepared different sizes of nanoparticles in pulsed laser ablation method. In this method, the particles size was tuned with increasing the laser pulse influence <sup>(47)</sup>. Yin *et. al.*, have prepared AgNPs of different sizes in microwave method using formaldehyde as reducing agent. Varying the concentration of AgNO<sub>3</sub> tuned the size of NPs <sup>(48)</sup>. Wan *et. al.*, have used reaction temperature as an important parameter for tuning the size of NPs <sup>(49)</sup>.

## CONCLUSIONS

Production of AgNPs of tunable sizes is a promising and interesting area of research in the field of nanoscience and nanotechnology. In this paper we have discussed about various physical, chemical and biological methods available for the size controlled synthesis of AgNPs. Despite to the methods available so far, it is still difficult and challenge to prepare AgNPs of desirable size because of their complicated growth mechanism and detailed chemistry involved. Hence

more research should be focused to understand the growth mechanism of size controlled metal NPs, which helps to establish the general understanding of the structures of different chemical reagents and biomolecules required to obtain NPs of desired size and shape. It is also necessary for the development of new low cost and nontoxic reproducible methods for the tuning the size of AgNPs. Additionally, future studies should focus on the remarkable properties of NPs with variation in their size and shape.

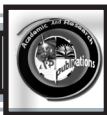
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# INCORPORATION OF ORGANOSELENIDES AND SELENOLACTAMS IN MICROEMULSIONS: Effect on Stability, Percolation and Size

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## ABSTRACT

A new methodology has been developed for the solubilization of organoselenides and organolactams by assimilating them in water/AOT+lecithin(LC)/isooctane mixed microemulsions. The stability of the system has been characterized by particle size and poly dispersity index measurements. The system has also been characterized for its spectral and physicochemical behavior indicating different type of water molecules associated with the surfactant head group and contributing to the formation of water pool. UV-vis absorption spectroscopy results indicate organoselenide moiety affects the bonding properties of the formulated microemulsion. The dynamics of the system has also been commented upon using conductivity measurements.

**Key Words :** Microemulsions, Organoselenides, Selenolactams, Physicochemical Behavior, Spectroscopic Studies and Thermodynamic Behavior.

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## INTRODUCTION

Aqueous solubility is one of the major indicators for solubility in intestinal fluids which serves as the pathway for various metabolic and bioavailability issues. A significant portion of biologically active organic compounds such as  $\beta$ -lactam and seleno- $\beta$ -lactam, suffer from the problem of poor water solubility, use of organic media for solubilization, easy degradation and low stability. Hence, one of the biggest challenges

these days is to devise new methods to enhance the solubility and stability of these compounds. Microemulsions are known to provide superior encapsulation vehicles for hosting such compounds. Although microemulsions/reverse micelles, formed in organic detergent solutions, are less interesting from a biological point of view than micelles or liposomes that form in water, they offer the advantage of a compulsory compartmentation. In fact, all water-soluble components added to the



hydrocarbon detergent system must go into the water pool of the reverse micelles—and in this sense reverse micelles provide one with a well-defined microreactor. Secondly, the flexibility of the microemulsion system offers an opportunity for dissolving molecules under certain conditions so that they themselves can 'choose' an optimal microenvironment for their functioning. Thirdly, because of their ability to control the size and properties of the water pool, the reverse micelles are interesting model candidates to mimic the water pockets that are often found in various bioaggregates such as proteins, membranes, and mitochondria. Fourthly, they not only encapsulate biomolecules but also protect them from degradation. Therefore, encapsulation in microemulsion results in the enhancement of solubility, stability, catalytic activity etc <sup>[1-4]</sup>. Certain enzymes are known to manifest enhanced activity at certain hydration levels <sup>[5]</sup>. However, the reports involving solubilization of water-insoluble organic compounds are few <sup>[6-9]</sup>. Mehta *et. al.*, <sup>[6-8]</sup> have recently carried out incorporation of pyrimidine and pyridine based organochalcogenides in microemulsion media. The effect of position of Se atom in the organoselenide on their encapsulation properties has been analyzed.

$\beta$ -lactam constitutes to be one of the most widely employed class of antibiotics. Another class of molecules that is of great interest is seleno- $\beta$ -lactams. The problem of ever-increasing bacterial resistance to  $\beta$ -lactam antibiotics poses a new challenge for the use of these antibiotics. Attempts to solve this difficulty have been made by exploring new  $\beta$ -lactam chemistry by the skeletal mod-

ification of naturally occurring  $\beta$ -lactam antibiotics <sup>[10]</sup>. However, the aspect of their solubility and stability in aqueous media is still unfamiliar and needs to be explored more.

Therefore, water/AOT+Lecithin/ isooctane system was chosen for the present study to elucidate the relationship between the microenvironments and the properties of water insoluble organoselenides/selenolactams. The present work has been devoted to study and understand the behavior of organoselenides and selenolactams in w/o microemulsion media from both experimental and theoretical point of view. Efforts have been made to assimilate organoselenides and selenolactams in AOT+Lecithin based microemulsion media. The choice of the system is based on the fact that the combination of anionic/cationic with a zwitterionic surfactant shows better water solubilization capacity as compared to single surfactant system. The results are expected to open a new gateway on the knowledge of organoselenide and organolactam interactions, stability in reversed micelles, which may be useful in designing synthetic routes for green synthesis and drug delivery applications of such synthetic lactams using microemulsion media <sup>[11,12]</sup>.

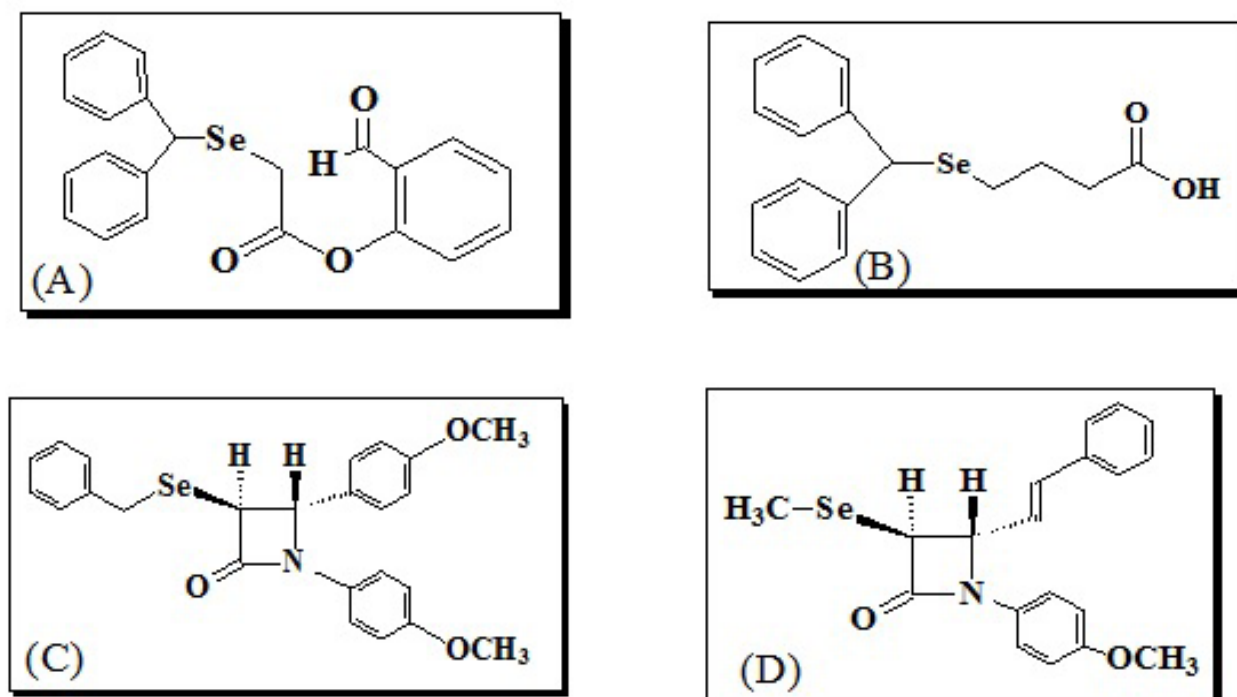
## Experimental

### MATERIALS AND METHODS

The preparation methodology involves the use of chemicals such AOT (Sodium bis(2-ethylhexyl) sulfosuccinate), LC (Lecithin), extracted from soybeans as surfactants obtained from Fluka and Sigma Aldrich with purity > 99% and > 95% respectively. The surfactants were used as supplied. Isooctane used was obtained from E-Mer-

ck with purity > 99%. Organoselenides including  $\text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_3\text{Se}$ ,  $\text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{Se}$ ,  $\text{Ph}_3\text{C}_6\text{H}_{10}\text{O}_3\text{NSe}$ ,  $\text{Ph}_2\text{C}_7\text{H}_{10}\text{O}_2\text{NSe}$  were crystalline solids synthesized in the laboratory and characterized through various spectroscopic techniques [13]. They

were used without further purification. The structures of assimilated organoselenenides have been presented in Scheme 1. Water used for sample preparation was triply distilled to get conductance less than  $3 \text{ Scm}^{-1}$ .



**Scheme 1** Structures of assimilated organoselenenides and selenolactams (A) 2-(diphenyl) methylselenoethanoate ( $\text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_3\text{Se}$ ) (B) 2-(diphenyl)methylselenobutanoic acid ( $\text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{Se}$ ) (C) trans-1-(4'-methoxyphenyl)-3-benzylseleno-4-(4'-methoxyphenyl)azetidin-2-one ( $\text{Ph}_3\text{C}_6\text{H}_{10}\text{O}_3\text{NSe}$ ) (D) trans-1-(4'-methoxyphenyl)-3-methylseleno-4-styrylazetidin-2-one ( $\text{Ph}_2\text{C}_7\text{H}_{10}\text{O}_2\text{NSe}$ ).

## METHODS

### Preparation of Samples

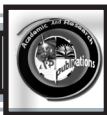
The microemulsions were prepared by weight. A mixture of AOT and LC ( $[\text{AOT}+\text{LC}] = 0.843 \text{ M}$  with  $[\text{AOT}]:[\text{LC}] = 0.70\text{M}:0.143\text{M}$  was prepared in 4 ml iso octane and then water was added dropwise to achieve the required  $\omega = [\text{H}_2\text{O}]/[\text{surfactant}]$  of 25. A series of experiments were carried out and  $\omega = 25$  was found to be the most acceptable to enhance assimilation of organoselenenides. All the samples were transparent and optically clear under the experimental condi-

tions reported. The detailed procedure for the formation of micro emulsion has been reported earlier [7].

### Particle Size Estimations

Particle size estimations (900 Optics) were carried out with Malvern Zeta Nano S 90 which calculates Z-average particle diameter using the Stokes Einstein equation assuming spherical droplets. For microemulsions, the droplet size was estimated by the average of three measurements. The samples were diluted times before measurement to avoid multiple scattering.





## Spectroscopic Measurements

### FTIR spectroscopy

FTIR spectra were recorded in the frequency range of  $4400 - 350 \text{ cm}^{-1}$  with the help of Perkin-Elmer (RX1) FTIR spectrometer using AgCl plates. For each sample 50 scans were recorded with the spectral resolution of  $2 \text{ cm}^{-1}$ . The data was repeated in triplicate to reduce the error, and one of the data was chosen for detailed study.

### Conductivity Measurements

Electrical conductivity measurements of the samples were carried out with PICO digital conductivity meter operating at 50 Hz from Labindia instruments with an absolute accuracy of  $\pm 3\%$  and precision of  $\pm 0.1\%$ . The cell constant used was  $1.0 \text{ cm}^{-1}$ . The temperature was kept constant with the help of RE320 Ecoline thermostat with an accuracy of  $\pm 0.01\text{K}$ .

For temperature percolation, experimental data were collected at different concentrations viz., 2.5, 5, 15 and 30 mM for  $\text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_3\text{Se}$ ,  $\text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{Se}$ ,  $\text{Ph}_3\text{C}_6\text{H}_{10}\text{O}_3\text{NSe}$  and  $\text{Ph}_2\text{C}_7\text{H}_{10}\text{O}_2\text{NSe}$  in the temperature range of  $30^\circ\text{C}$  to  $-50^\circ\text{C}$ . The composition of the microemulsion was kept constant at with  $[\text{surfactant}] = 0.743$  and  $[\text{AOT}]:[\text{LC}] = 0.70\text{M}:0.143\text{M}$  and  $\omega = 25.0$ . All the samples were prepared by weight and were transparent and optically clear under the conditions of conductivity reported.

## RESULTS AND DISCUSSION

### Particle size and stability studies

Assessment of the literature<sup>[14]</sup> depicts that AOT+Lecithin droplets are usually ellipsoidal so identifying the spatial size distribution of the system becomes difficult. As a result the projected profiles are no longer uniform and an analytical solution is not possible. So the data obtained is apparent<sup>[15]</sup>. However, it has been reported by<sup>[16]</sup>

that on dilution, the interparticular interactions are minimized and the droplet structure can pass from a reversed spherical droplet to a reversed rod-shaped droplet, hexagonal phase, lamellar phase, cubic phase and various other structures until, after appropriate dilution, a spherical droplet will be formed again. So the particle size measurements of the reverse micelle with and without the addition of organoselenides have been carried out with dilution to minimize the scattering effects assuming spherical droplets. The results have been tabulated in Table 1 with representative plot of  $\text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_3\text{Se}$  shown in Fig. 1. An increase in the mean diameter of reverse micellar droplet with the addition of organoselenides has been observed. The size follows the order: without additive  $< \text{Ph}_3\text{C}_6\text{H}_{10}\text{O}_3\text{NSe} < \text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_3\text{Se} < \text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{Se} < \text{Ph}_2\text{C}_7\text{H}_{10}\text{O}_2\text{NSe}$ . The droplet size observed in the present case is also higher and this is possible if one considers the formation of non spherical structure in the microemulsion with large effective diameters<sup>[17,18]</sup>. The observed PDI values lies in the range of 0.38-0.46. Evaluation of the sample stability after 15 days indicate a slight increase in size and PDI values. This indicates that net attractive forces hydrophobic or vanderwaals are stronger than the repulsive forces and hence the droplets tend to flocculate. However no visible evidence of gravitational separation or creaming was observed during the storage period. The observed trend is opposite to the conductivity data, where the larger droplet size is considered to favor percolation. This can be explained by considering different mechanisms of the two measured processes. As percolation, apart from depending upon droplet size, depends on the number of other factors such as feasibility of formation of transient tube, effective overlap etc. have to be taken into consideration to explain the process completely.



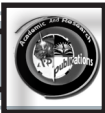
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**Table 1. Estimated diameter of the droplets at = 25 effected by the addition of organoselenides/selenolactams = 5mM**

Organoselenides	Particle size (nm)	PDI	stability (after 15 days)	PDI
Without	168.6±20	0.38±0.021	188.6±10	0.339
$\text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_3\text{Se}$	210±11.5	0.28±0.054	245±25	0.665
$\text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{Se}$	231.5±26	0.46±0.062	260.5±30	0.589
$\text{Ph}_3\text{C}_6\text{H}_{10}\text{O}_3\text{NSe}$	200.4±15	0.29±0.001	230.4±12	0.444
$\text{Ph}_2\text{C}_7\text{H}_{10}\text{O}_2\text{NSe}$	298.7±10	0.38±0.028	325.7±18	0.586

Molar Solubilization Ratio( $\chi$ ) and micelle/aqueous phase partitioning of organochalcogens (K)



Solubilization of a substance can be estimated using three parameters, the molar solubilization ratio ( $\chi$ ) and the micelle-water partition coefficient ( $K$ )<sup>[19]</sup> and oil/water partition coefficient ( $P$ ). The oil /water partition co-efficient was determined by dissolving 2mg organoselenide/selenolactam in isooctane. Water was then added in 1 : 1 ratio(v/v). The mixture was shaken for 10 min and centrifuged for 1 h. The two layers were separated and the content of organoselenide in aqueous layer was assayed by UV-visible spectrophotometer. The final content of organoselenide/selenolactam in the lipophilic phase was calculated by subtracting the content of organoselenide/selenolactam in aqueous phase from initial loaded content of organoselenide in lipophilic phase. Further, the effect of presence of surfactant on the partition of organoselenide/selenolactam in oil/water was also studied. The value of partition coefficient in Oil/water system are given in Table 2.

The value is characterized as the amount of solute (organoselenide/selenolactam) that can be solubilized by one mole of micellar surfactant. It illustrates the ability of the surfactant to solubilize the organoselenide and is given by

$$\chi = \frac{\{[S_t] - [S_{cmc}]\}}{[C_t - cmc]}$$

where  $[S_t]$  is the total apparent solubility of the solubilize,  $[S_{cmc}]$  is the apparent solubility of the organoselenide at cmc, which is taken as their water solubility,  $[S]$ , because it changes only very slightly up to the cmc of the surfactant.  $C_t$  is the molar concentration of surfactant in so-

lution and **cmc** is the **critical micelle concentration**. is equal to the ratio of organoselenide concentration solubilized in micelles to the surfactant concentration in the micellar form and is obtained from the slope of the curves that results when solubilize concentration is plotted against the surfactant concentration. The variation of solubilities for  $\text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_3\text{Se}$ ,  $\text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{Se}$ ,  $\text{Ph}_3\text{C}_6\text{H}_{10}\text{O}_3\text{NSe}$ ,  $\text{Ph}_2\text{C}_7\text{H}_{10}\text{O}_2\text{NSe}$  are plotted in Fig 2.

The aqueous solubilities of organoselenides/selenolactams compounds amplify linearly with surfactant concentration, demonstrating their solubility enrichment over that in water. This phenomenon is presumably associated to the micellar solubilization. The values from the above plots are tabulated in Table 2 for the added organoselenides and selenolactams respectively. To further examine the efficiency of solubilization, the micelle-water partition coefficient,  $K$ , of the solubilize flanked by the micelle and aqueous phases has also been evaluated<sup>[20]</sup> via. equation 2.

$$K = \frac{\{[S_t] - [S]\}}{[S]}$$

The obtained value of  $\text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{Se}$ ,  $\text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_3\text{Se}$ , are higher than  $\text{Ph}_2\text{C}_7\text{H}_{10}\text{O}_2\text{NSe}$ ,  $\text{Ph}_3\text{C}_6\text{H}_{10}\text{O}_3\text{NSe}$ . The low value of Log  $P$  observed with the introduction of surfactant indicates the presence of organoselenide at the interface. This also indicates that the intrinsic solute properties and its specific interactions with the micellar pseudo-phase contribute to the distribution of organodiselenide between the two pseudophases.



The obtained value of  $\text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{Se}$ ,  $\text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_3\text{Se}$ , are higher than  $\text{Ph}_2\text{C}_7\text{H}_{10}\text{O}_2\text{NSe}$ ,  $\text{Ph}_3\text{C}_6\text{H}_{10}\text{O}_3\text{NSe}$ . The low value of Log P observed with the introduction of surfactant indicates the presence of organoselenide at the inter-

face This also indicates that the intrinsic solute properties and its specific interactions with the micellar pseudo-phase contribute to the distribution of organodiselenide between the two pseudo-phases.

**Table 2 Molar Solubilization Ratio ( $\chi$ ) and micelle-water partition coefficient (K) for  $\text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_3\text{Se}$ ,  $\text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{Se}$ ,  $\text{Ph}_3\text{C}_6\text{H}_{10}\text{O}_3\text{NSe}$ ,  $\text{Ph}_2\text{C}_7\text{H}_{10}\text{O}_2\text{NSe}$  using AOT+LC as surfactant**

Organodiselenide	$\chi$	K(mM dm <sup>-3</sup> )	Log P
$\text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_3\text{Se}$ ,	0.46	1.006	2.15
$\text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{Se}$	0.52	3.001	4.26
$\text{Ph}_3\text{C}_6\text{H}_{10}\text{O}_3\text{NSe}$	0.39	0.991	2.92
$\text{Ph}_2\text{C}_7\text{H}_{10}\text{O}_2\text{NSe}$	0.31	0.776	1.69

## Spectroscopic Measurements

### FTIR spectroscopy

In any reverse micellar formulation, the information about the water pool is obtained from the OH band [21]. It has already been reported by Mehta *et. al.*, [22] that the water close to interface exhibits behavior markedly different from that of bulk water as it processes restricted mobility and a depressed freezing point and lacks the normal hydrogen-bonded structure present in the bulk [23]. As this water is composed of four different states, viz., bound water (two types), trapped water, and free water, it is reasonable to assume that the total peak area corresponding to the water band is the sum of the peak areas of the different states of water [24].

Thus, total peak area is given by

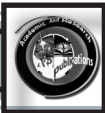
$$G_{\text{Total}} = G_1 + G_2 + G_3 \quad \dots\dots\dots (3)$$

If  $P_F$  is the fraction of free water corresponding to the  $3290 \pm 20 \text{ cm}^{-1}$  peak,  $P_B$  is the fraction of bound water corresponding to the  $3490 \pm 20 \text{ cm}^{-1}$  peak, and  $P_T$  (water bound to the surfactant head group and counterion) is the fraction of trapped water corresponding to the  $3610 \pm 10 \text{ cm}^{-1}$  peak, then their respective values can be calculated from the following relations

$$P_F = \frac{G_1}{G_{\text{TOTAL}}} \quad P_B = \frac{G_2}{G_{\text{TOTAL}}} \quad P_T = \frac{G_3}{G_{\text{TOTAL}}}$$

Figure 3 depicts the changes occurring in OH band by the addition of organoselenide/selenolactam at  $\omega = 25$  and the data has been tabulated in Table 3. Deconvolution of the band shows four sub peaks revealing different kinds of water molecules in one band. The peak for free water has been found to show blue shift with the incorporation of organoselenide





and selenolactam indicating the presence of the assimilated entity at the micellar interface. This further indicates H-bonding interactions among the water molecules are replaced by organoselenide / selenolactam and OH of water H-bonds. Other contributions to the shift can arise from structural disorder within the H-bonded molecular cluster confined within the micellar core. Attempts have also been made to calculate the number of water molecules associated with a particular moiety at the interface. However, no regular trend can be generalized in this case as the added organoselenide/organolactam has its own characteristics and this effect the binding with a particu-

lar site. Also with the increase in  $\omega$  a shift towards higher wavenumber is observed. (Representative plots for variation of  $\omega$  for  $\text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_3\text{Se}$  and  $\text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{Se}$  have been shown in Fig I supplementary material). The change becomes constant after the shoulder point i.e.  $\omega = 15$  is reached. This indicates that on initial addition upto  $\omega = 15$ , water goes to hydrate the interface of the reverse micelle. However, any further addition just adds the water to the core of the microemulsion system. The stretching frequency follows the trend without  $< \text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{Se} < \text{Ph}_3\text{C}_6\text{H}_{10}\text{O}_3\text{NSe}$ . This indicates significant interaction between the added organoselenide/selenolactam and water pool.

**Table 3. Positions obtained by from FTIR stretching bands  $\omega = 25$  in the presence organoselenides/selenolactams**

Organoselenide	water with sodium counterion	free water	bound water in water pool hydrogen to sulfo-group of AOT	trapped water in the palisade layer
Without	3251.25,0.258	3320.87,0.325	3421.25,0.2569	3498.27,0.158
$\text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_3\text{Se}$	3356.07, 0.322	3137.61, 0.356	3507.05, 0.065	3612.10,0.262
$\text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{Se}$	3361.35,0.296	3231.89,0.352	3487.71,0.258	3592.69,0.094
$\text{Ph}_3\text{C}_6\text{H}_{10}\text{O}_3\text{NSe}$	3385.06,0.179	3271.88,0.356	3399.56,0.228	3529.96,0.268
$\text{Ph}_2\text{C}_7\text{H}_{10}\text{O}_2\text{NSe}$	3405.20,0.268	3189.57,0.359	3498.24,0.289	3591.16,0.084

Also the peak centered in the region  $1700\text{-}1730\text{ cm}^{-1}$ , assigned to CO stretching shows similar trend of shift towards the lower wavenumber with the incorporation of organodiselenide. (Table I Supplemenatry material). A shift of around  $1.03, 1.45, \text{ cm}^{-1}$  is observed for  $\text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_3\text{Se}$ ,  $\text{Ph}_2\text{C}_3\text{H}_4\text{O}_2\text{Se}$  compared to pure microemulsion. Significant splitting is seen in the CO spectra of

$\text{Ph}_3\text{C}_6\text{H}_{10}\text{O}_3\text{NSe}$  and  $\text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{NSe}$ . This can be due to the fact that the assimilated Lactams have active N end that disrupts the electric field produced by the charged head groups of the surfactant resulting in different interactions between the selenolactam and the surfactant head groups. Similar splitting is seen in the peaks COC, and  $\text{PO}_4^{3-}$  peaks of the two lactams as compared to



other compounds. However, no shifts were seen in the spectra of  $\text{SO}_3^-$  reveal the entrapping of organoselenide in the micellar interface. The results further depict the presence of specific interactions between the interfacial water molecules and the organoselenide moieties. The presence of organoselenides perturbs the H-bonding capabilities of the OH, CO and C-O-C groups of the surfactant molecules. Significant changes can be seen in CH spectra indicating the presence of the organoselenides in the continuous medium ruling out the presence of interactions of the organoselenide with this part of the AOT molecule.

## Conductivity Measurements

The variation conductivity with temperature measured at different organoselenide concentrations has been presented in Fig. 4. The threshold temperature  $\theta_c$  has been determined from  $d\log/d$  vs  $\theta$ , where maxima corresponds to percolation threshold. Usual procedure reported earlier by the authors has been followed [6-8]. The threshold values of  $\theta_c$  at different concentrations have been listed in Table 4. The results depict that  $\theta_c$  increase with the increase in concentration of the added organoselenide/selenolactam. The value of  $\theta_c$  for all concentration under study varies as  $\text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{Se} \sim \text{Ph}_2\text{C}_7\text{H}_{10}\text{O}_2\text{NSe} < \text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_3\text{Se} < \text{Ph}_3\text{C}_6\text{H}_{10}\text{O}_3\text{NSe} < \text{without additive}$ . The Sigmoidal Boltzmann fitting (SBE) procedure reported by Hait *et. al.*, [25] has also been employed to get the percolation threshold. Fitted curves of conductance - have been depicted in Fig. 4.

The efficacy of additive has been cal-

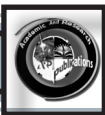
culated using Eq. (5) as reported in [25,26]

$$\theta_c^a = \theta_c^0 + S_k C$$

Where  $\theta_c^0$  and  $\theta_c^a$ , C have their usual meaning. Eq. (5) has been used to calculate the  $\theta_c$  data of systems listed in Table 4. The values of the slope ( $S_k$ ) obtained have also been included in table 4. On the basis of values of  $S_k$ , the efficacy of the additive in favoring percolation follows the order:  $\text{Ph}_2\text{C}_7\text{H}_{10}\text{O}_2\text{NSe} > \text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{Se} > \text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_3\text{Se}$ . Species yielding negative slopes are percolation assistants, and solutes yielding positive slopes are percolation retardants [26].

The mutual contact and bridging between droplets is due to the presence of additives either at the interface (e.g., hydrotopes and bile salts) [27] or in the core of the droplets (e.g., poly (ethylene glycol) s) [28]. The results depict that as a result of low water solubility the added organoselenide/selenolactam molecules are restricted mainly in the surfactant film. This results in the straight bridging of nanodroplets in case of  $\text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{Se}$  and  $\text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_3\text{Se}$  and thus the channel formation is favored at higher organoselenide concentration whereas at low concentration the bridging is not much efficient. However, selenolactam;  $\text{Ph}_3\text{C}_6\text{H}_{10}\text{O}_3\text{NSe}$  and  $\text{Ph}_2\text{C}_7\text{H}_{10}\text{O}_2\text{NSe}$  contains active N-end, which helps the, the molecule adheres to the droplet interface. This configuration can result in the eclipsed bridging of nanodroplets due to cyclization but this favors the percolation process. With the increase in concentration of organoselenide/selenolactam, the interfacial rigidity increases and this reduces the droplet association thereby increasing the percolation threshold.

However, the results are contrary to oth-



er reports <sup>[6,7]</sup> published on organoselenides where the percolation process is delayed. This is because of the steric hindrance offered by  $C_{10}H_6N_2Br_2Se_2$  and  $C_{10}H_4N_2Br_4Se_2$  <sup>[6,7]</sup> due to the presence of two and four bromine atoms/organoselenide is very high as compared to the phenyl /  $\beta$  Lactam ring in the present case. As a result of which straightforward bridging is observed whereas in the present case the bridging is somewhat staggered. Therefore an efficient intradroplet contact is established which results in material exchange. Similar results have been observed by Moulik *et. al.*, <sup>[27]</sup> to explain the effect of hydrotopes on droplet percolation.

The activation energy for conductance percolation has been estimated by using Arrhenius equation. The calculated  $E_p$  <sup>[29,30]</sup> values, using do not follow a specific trend. The results are also contrary to the earlier reported results <sup>[27]</sup>, which depicts that percolation assisting derivatives should have higher  $E_p$  values. A plausible explanation for these results may be put forward as argued by Moulik *et. al.*, <sup>[27]</sup> for the systems AOT/hydrocarbon/water in the presence of aromatic methoxy hydrotopes. The fission of the

bulky droplets into a pair of singlets with associated transfer of counter ions required high activation energy. The bridging of the droplet with an efficient binding additive may reduce  $\theta_c$  but it is the process of separation that decides the magnitude of  $E_p$ . The magnitude of  $E_p$  is thus essentially controlled by the separability of the droplets from the “fused” to the “free” state. This is because of different structures and physicochemical properties of the added moieties in comparison to the previous studies. Bulkier organoselenide/selenolactam molecules redisperse around the droplets associated with the transport of the  $Na^+$  ion. The activated state of mass transfer thus yields higher  $\Delta E_p$ . The value of  $\Delta E_p$  follows an intriguing correlation with  $\theta_c$  for the respective set of systems. The phenomenon of percolation is essentially different from the normal mode of conduction, and the magnitude of  $\Delta E_p$  largely depends on the separability of the droplets. This is because of different structures and physicochemical properties of the added moieties in comparison to the previous studies. Also in addition to clustering, fusion, mass transfer, and fission, there may be other steric and interacting factors for the process of percolation.

**Table 4. Percolation threshold and activation energy parameters for temperature induced percolation of water/AOT+LC/Isooctane microemulsion system with varying concentrations of organoselenide/selenolactam at  $\omega = 25$ .**

Organoselenide	[Conc.] (mM)	$\log \sigma_i$	$\log \sigma_f$	$\theta_c(K)$		$\Delta E_p$ (KJ/mol)	$-S_k$
				differential	SBE		
Without	-	-0.60±0.04	2.93±0.06	316.80	316.80±0.09	-	
$Ph_2C_9H_{10}O_3Se$	2.5	-0.43±0.05	3.01±0.06	313.15	312.47±0.01	28.4	0.21±0.04
	5	-0.55±0.04	3.03±0.05	311.16	313.70±0.07	26.8	
	15	-0.96±0.06	3.05±0.04	315.17	314.67±0.09	30.3	
	20	-0.79±0.02	3.06±0.02	315.78	315.78±0.10	26.1	
$Ph_2C_5H_8O_2Se$	2.5	-0.55±0.05	3.29±0.12	312.14	311.89±0.17	45.7	0.29±0.02
	10	-0.50±0.05	2.96±0.06	312.19	312.32±0.14	25.5	
	20	-0.59±0.04	3.11±0.06	317.11	316.20±0.10	25.9	
$Ph_3C_6H_{10}O_3NSe$	2.5	-0.74±0.02	3.02±0.04	315.15	315.53±0.08	29.2	-
$Ph_2C_7H_{10}O_2NSe$	5	-0.71±0.02	3.05±0.04	314.15	314.75±0.10	28.6	0.40±0.03
	10	-0.50±0.03	3.05±0.03	316.15	314.57±0.09	27.6	

**Thermodynamics of droplet clustering, association mode**

For percolation to occur droplet clustering is an essential feature. It is governed by various factors such as droplet density, size and the type of additive. The process reported by Moulik group [25,27] and Alexandridis *et. al.*, [31] states that the dilution of percolating microemulsion lowers the conductivity rapidly until the clusters dissociate into individual droplets below  $\theta_c$ . The phenomenon corresponds to the process of demicellization when the surfactant concentration is lowered below the **critical micellization concentration (cmc)**. The standard free energy,  $G_{cl}^0$  and the values of thermodynamic parameters have been calculated using standard equations

$$\Delta G_{cl}^0 = RT \ln X_d \quad \text{.....(6)}$$

where  $X_d$  is the mole fraction of the droplets corresponding to the percolation threshold. Estimated values of  $G_{cl}^0$  for different concentrations for temperature induced percolation are listed in Table 5. The negative value of  $G_{cl}^0$  indicates spontaneous formation of clusters. The results further reveal a more negative value of  $G_{cl}^0$  in the presence of organoselenide/selenolactams.

Application of Gibbs - Helmholtz equation

allows calculating the standard enthalpy of cluster formation,  $H_{cl}^0$

$$\Delta H_{cl}^0 = \partial (\Delta G_{cl}^0 / T_c) / \partial (1/T_c) \quad \text{.....(7)}$$

and finally the standard entropy of cluster formation per mole of the droplet,  $S_{cl}^0$  is given by the relation

$$\Delta S_{cl}^0 = (\Delta H_{cl}^0 - \Delta G_{cl}^0) / T_c \quad \text{.....(8)}$$

In order to estimate  $H_{cl}^0$ ,  $G_{cl}^0$  is plotted as a function of inverse percolation temperature and the slope of  $G_{cl}^0 / \theta_c$  vs  $1/T_c$  plot yields the value of  $H_{cl}^0$  (Fig. 5). The value has been obtained by least square curve fitting. The obtained positive values of  $H_{cl}^0$  and  $S_{cl}^0$  accounts for a strong heat-absorbing step in the percolation process. The values of  $H_{cl}^0$  and  $S_{cl}^0$  follow the order: without >  $\text{Ph}_3\text{C}_6\text{H}_{10}\text{O}_3\text{NSe}$  >  $\text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_3\text{Se}$  >  $\text{Ph}_2\text{C}_7\text{H}_{10}\text{O}_2\text{NSe}$  >  $\text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{Se}$ . The low positive enthalpy change in the presence of selenides than in the absence indicates a relatively less organized i.e. easily disruptable micro surrounding of the droplets of  $\text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{Se}$  and  $\text{Ph}_2\text{C}_7\text{H}_{10}\text{O}_2\text{NSe}$  whereas it is more organized in  $\text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_3\text{Se}$  and  $\text{Ph}_3\text{C}_6\text{H}_{10}\text{O}_3\text{NSe}$  as compared to the system without organoselenide.

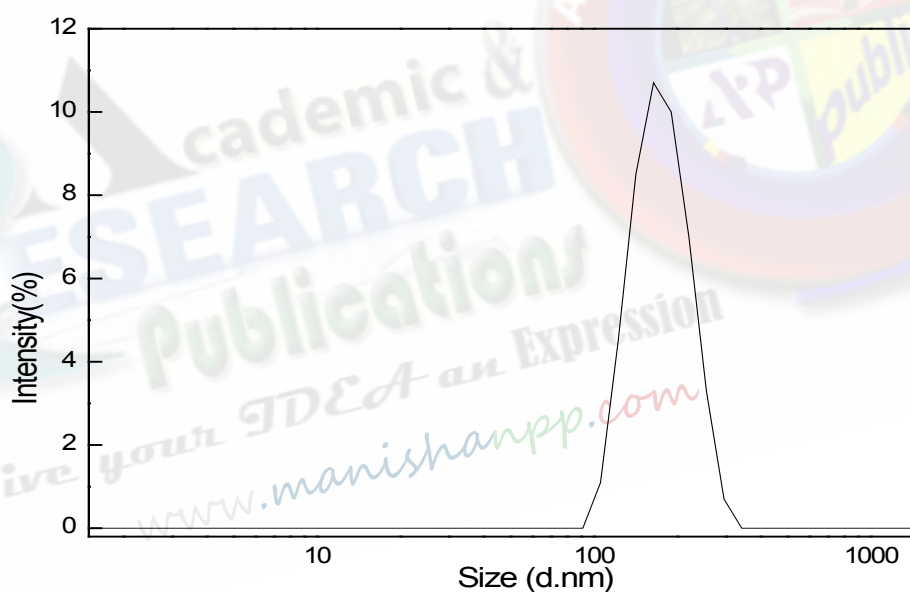




**Table 5. Energetic parameters of droplet clustering for water/AOT+LC/Isooctane microemulsion at  $\omega = 25$ , Organoselenide = 3 mM and varying  $S = [\text{oil}]/[\text{surfactant}]$**

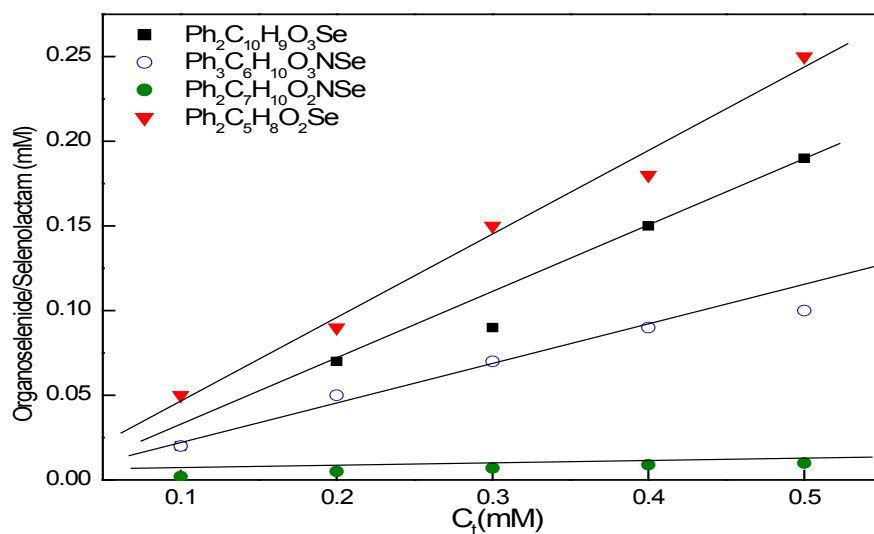
Organoselenide	$S=[\text{Oil}]/[\text{AOT}]$	$-\Delta G^0_{cl}/\text{kJmol}^{-1}$	$\Delta H^0_{cl}/\text{kJmol}^{-1}$	$\Delta S^0_{cl}/\text{JK}^{-1}\text{mol}^{-1}$
Without	5	22.66	$136.56.2 \pm 7.5$	516.0
	7	23.82		
	9	25.96		
	15	24.14		
$\text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_3\text{Se}$	5	22.82	$63.13 \pm 5.9$	277.2
	7	24.04		
	9	24.92		
	15	26.86		
$\text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{Se}$	5	23.16	$55.1 \pm 5.8$	226.4
	7	23.84		
	9	24.64		
	15	27.01		
$\text{Ph}_3\text{C}_6\text{H}_{10}\text{O}_3\text{NSe}$	5	22.80	$75.83 \pm 5.8$	316.23
	7	23.05		
	9	24.13		
	15	26.69		
$\text{Ph}_2\text{C}_7\text{H}_{10}\text{O}_2\text{NSe}$	5	22.89	$57.42 \pm 6.1$	258.42
	7	23.97		
	9	24.92		
	15	27.01		

## Figures

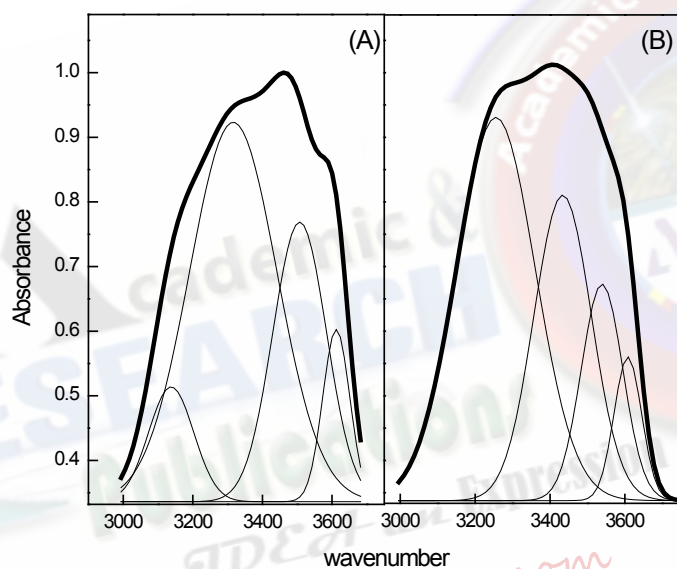


**Fig. 1. Estimated droplet size of  $\text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_3\text{Se}$ .**

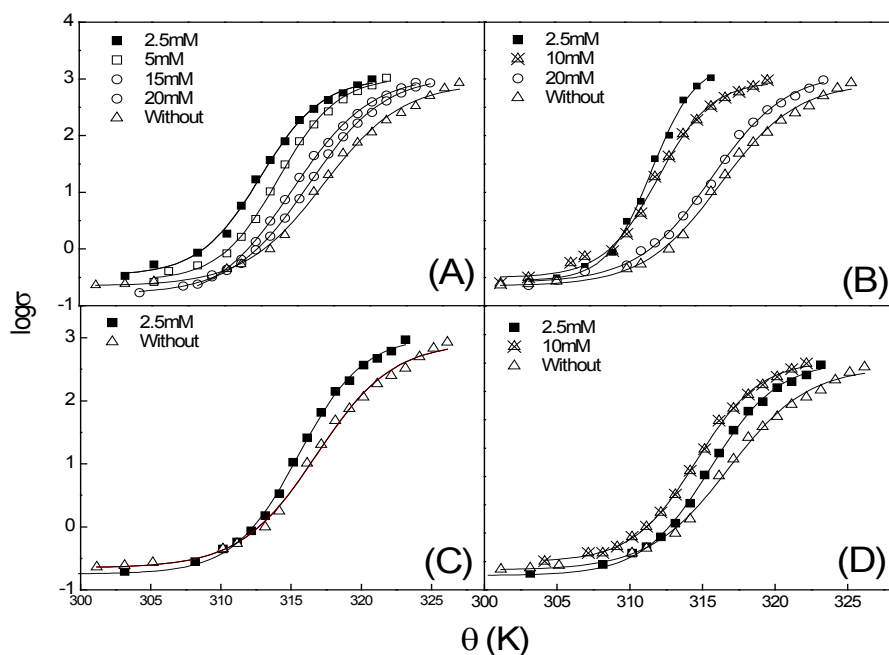




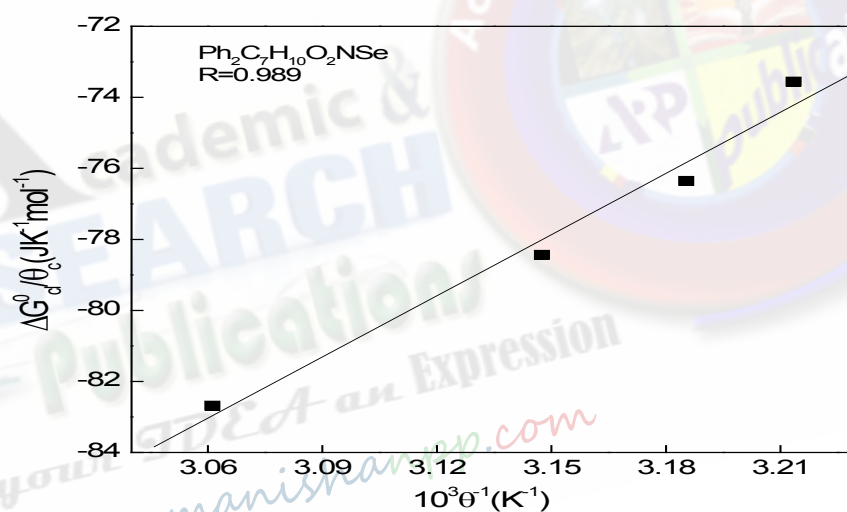
**Fig.2. Variation of Solubility of added organoselenides/selenolactams with total surfactant concentration (C<sub>t</sub>).**



**Fig. 3. Deconvoluted FTIR spectra of water/AOT/LC/isooctane at  $\omega = 25$  (A) Ph<sub>2</sub>C<sub>9</sub>H<sub>10</sub>O<sub>2</sub>Se (B) Ph<sub>2</sub>C<sub>3</sub>H<sub>4</sub>O<sub>2</sub>Se.**



**Fig. 4. Variation of specific conductance with temperature for different organoselenides (A)  $\text{Ph}_2\text{C}_9\text{H}_{10}\text{O}_2\text{Se}$  (B)  $\text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{Se}$  (C)  $\text{Ph}_3\text{C}_6\text{H}_{10}\text{O}_3\text{NSe}$  (D)  $\text{Ph}_2\text{C}_7\text{H}_{10}\text{O}_2\text{NSe}$  (solid lines show SBE fitting).**

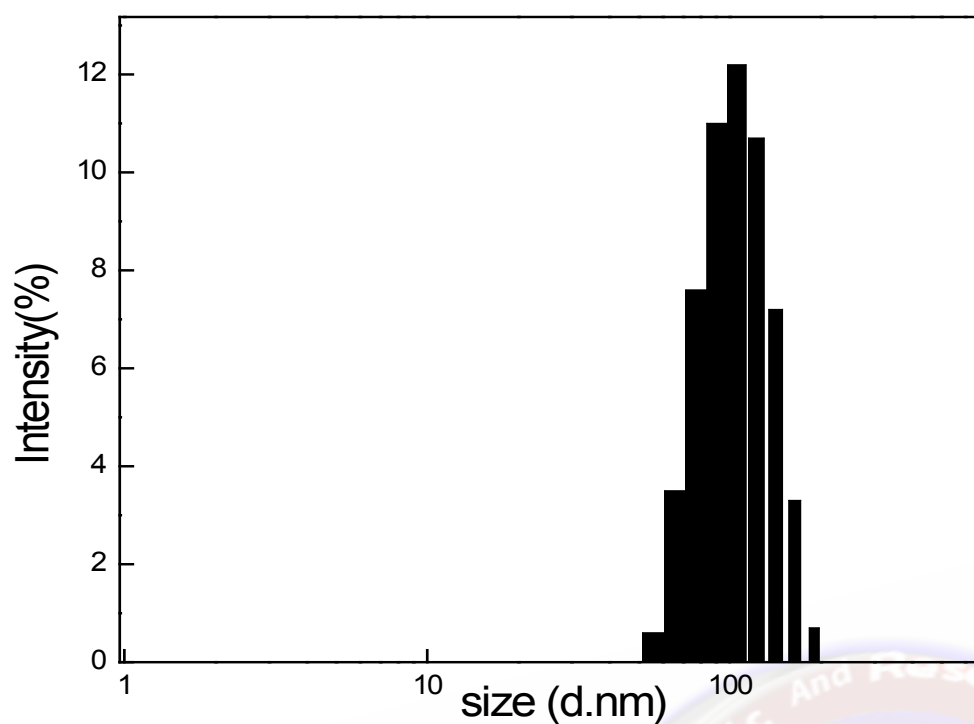


**Fig. 5.  $\Delta G^\circ / \theta_c$  as a function of  $\theta^{-1}$  to evaluate  $\Delta H^\circ$  in the presence of  $\text{Ph}_2\text{C}_5\text{H}_8\text{O}_2\text{NSe} = 5 \text{ mM}$  and  $\omega = 25$ .**



### Graphical Abstract

A new methodology has been developed for the stabilization of organoselenides/selenolactams by incorporating them in mixed surfactant microemulsion system.



**Table I** Positions obtained by from FTIR stretching bands water/AOT+LC/isooctane microemulsion at  $\omega = 30$  in the presence organoselenides

Organoselenide	CO (cm <sup>-1</sup> )	SO <sub>3</sub> (cm <sup>-1</sup> )	COC (cm <sup>-1</sup> )
Without	1735.88	1047.07	1213.74,1246.25
Ph <sub>2</sub> C <sub>9</sub> H <sub>10</sub> O <sub>2</sub> Se	1730.85	1047.07	1213.32,1246.01
Ph <sub>2</sub> C <sub>5</sub> H <sub>8</sub> O <sub>2</sub> Se	1732.3	1047.06	1225.5,1236.22
Ph <sub>3</sub> C <sub>6</sub> H <sub>10</sub> O <sub>3</sub> NSe	1711.2,1729.42,1767	1047.71	1211.5,1234.7,1230
Ph <sub>2</sub> C <sub>7</sub> H <sub>10</sub> O <sub>2</sub> NSe	1697,17102,1720.8,1730.5	1046.34	1211.90,1238.26,1224.07



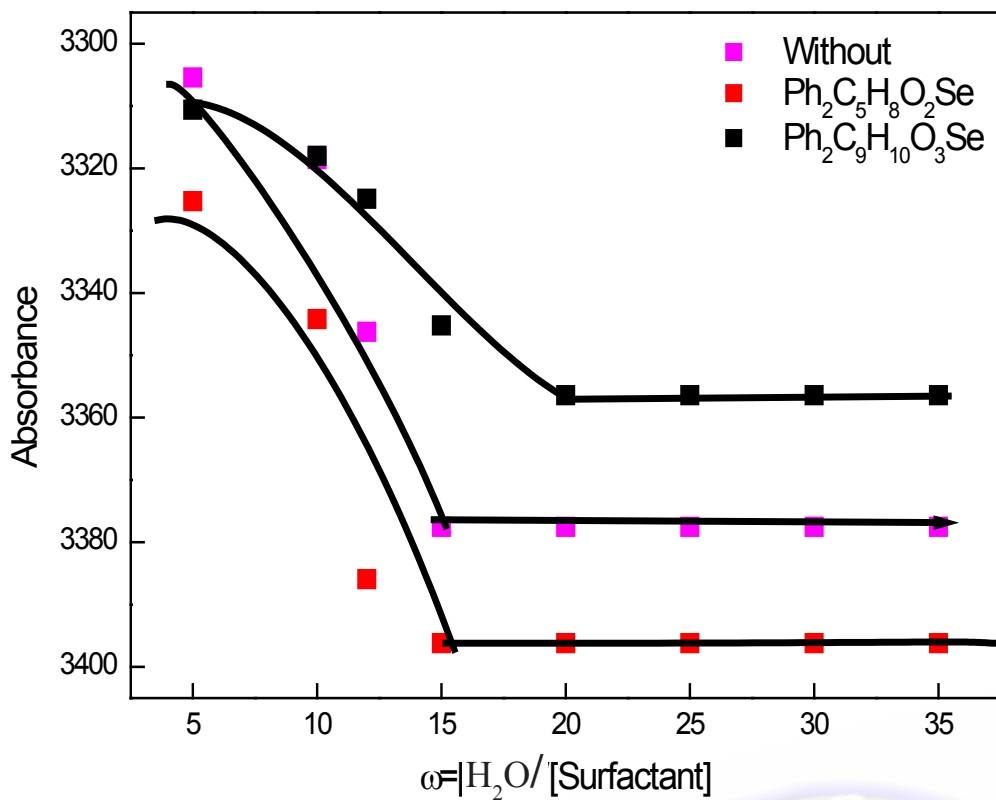


Fig. I. FT IR spectra of -OH functionality as a function of water/surfactant molar ratio



## ACKNOWLEDGEMENT

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## CONCLUSIONS

In conclusion, we have detected that the incorporation of organoselenides/selenolactams in microemulsions significantly affects the water pool properties and the electric percolation phenomenon. Knowledge of the mixed surfactant systems could have many applications in material synthesis. The expanded range of micellar geometries and functionalities available in mixed surfactant systems not only makes them useful for applications but also provides opportunities for fundamental studies of self assembly. The rationale for using microemulsions as a medium for solubilization of organoselenides is to provide alternative route for solubilization of these compounds and thus help in organic synthesis. Conductivity and spectroscopy results indicate the presence of specific interactions between organoselenides, AOT and Lecithin and water molecules and the location of the organoselenide at the interface. FTIR results also indicate the presence of four types of water molecules. The clustering of the microemulsion droplets is shown to be endothermic. The microenvironment of the system in the absence of selenolactams is found to be more organized than in their presence.

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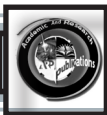


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## LETTER TO THE EDITOR

### Let's Talk Nanotechnology : Some Applications and Scope For Future Research

**Dr. Kavyanjali Shukla**

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Dr. Kavyanjali Shukla holds a PhD. in Zoology. She is an author, researcher, an avid science communicator and an environmentalist who has a keen interest in volunteering for various social causes. She is also working as **Coordinating Editor, International Journal of Nanoscience and Technology (IJNST) and Geosciences (IJGST), Academic & Research Publications house, India.**

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September, 1, 2014, I got the opportunity to deliver a science talk among the young and enthusiastic students of Christ University, Bangalore. The topic of discussion was " *The fundamentals and applications of Nanoscience and technology- an overview; subtopic : possible application of nanotechnologies in amoebic keratitis.* " I started off as how we used to study science during the early, historical times to what and how we study it today. How the methods of studying science have really evolved, with each revolutionary discovery and how we have used our past knowledge to decipher new techniques for further studies. Nanoscience is the study of controlling matter at an atomic and molecular scale. Generally nanotechnology deals with structures sized between 1 to 100 nanometer and involves developing or modifying materials or devices within that size. Prefix 'nano' is derived from the Greek word meaning dwarf. One nanometer (nm) is equal to one-billionth of a meter,  $10^{-9}$  m. By definition of Hilary Thomson, 2008, "*Nanotechnology has been called a molecular revolution—innovation so profound it will allow us to rebuild our world molecule by molecule. The unprecedented benefits of such control over matter have the potential to permeate every aspect of our lives. But so do the risks.*" The term 'nanotechnology' was not used until 1974, when Norio Taniguchi, a researcher at the University of Tokyo, Japan used it to refer to the ability to engineer materials precisely at the nanometre level. We often use two terms, Nanoscience and nanotechnology, here, we may understand nanoscience as the study of phenomena and manipulation of materials at atomic, molecular and macromolecular scales, where properties differ significantly from those at a larger scale, while, Nanotechnology is more of a phenomenon that enables the designing, production of structures, devices and systems that have novel applications. This is done by controlling the size and shape of the material at the nanometer ( $10^{-9}$  m of a meter) scale technology.

It is now, that we have to understand that nanotechnology might be our very near future. In order to understand it, we need to be first acquainted by it. What is nano science? Why we have to know about it ? What benefits do we derive from studying



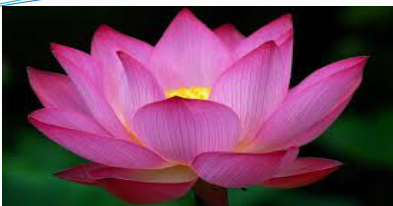
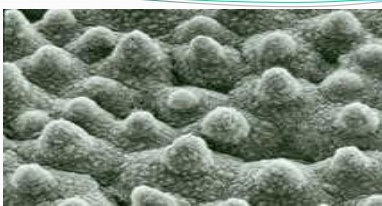

it, these all queries must be flashing in our minds and these will be dealt in details in order to clear our doubts. But, let us first understand the objectives of the present discussion. As of now, I have found two objectives that I discussed among the undergraduate students of Christ university. These are as follows:

- To realize that nanotechnology is our future and so to discuss its developments and applications and
- To spread awareness regarding the need of knowledge upgradation for future career prospects and career goals.


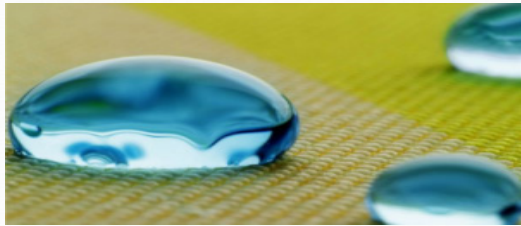
Let's acknowledge the fact that nanotechnology or nanoscience have always existed in nature. It is only that now we are discovering it and inventing novel things by deriving inspiration from these living beings that are a result of a phenomenon called adaptation and being favored by nature.

One such example is of a lotus flower. The leaves of lotus flower, *Nymphaea* sp. is waterproof. The reason behind this waterproof quality is the presence of very small (nano projections) throughout the surface. So, when water falls on a leaf surface, the droplets simply sits on these enormously small projections and then rolls off through the entire surface taking along with it, all the dirt as well. Now, this same technique have been borrowed from nature and some special nano fibres have been developed for cloth making, that have the properties of being waterproof and dirt resistant. Such a cloth is now utilized for making garments such as shirts, which are now also, readily available in the market. Such garments are water and dirt resistant.

**Nanotechnology exists in nature since time immemorial**

		
Lotus petals and leaves are water proof because of nanostructures	Electron microscopic view of lotus leaves	How a water droplet rolls on a leaf, taking of all the dirt

**The same principle is now utilized for making of nano fibres for cloth making, such clothes will be waterproof and will not get dirty by sweat and dirt.....its ALREADY IN THE MARKET**

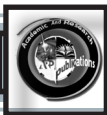
	
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Another, such example is that of a water striders, that have remarkable non-wetting legs that enable them to stand and move quickly on water. Each leg is covered with large numbers of angled tiny hairs (microsetae) with tiny nanogrooves. Air is trapped in spaces in the microsetae and nanogrooves to form a cushion where the leg touches the water. This prevents the leg from getting wet. So, now, we may understand how these naturally occurring nanostructures have actually inspired the modern day scientists for novel inventions. Besides, there are many such examples of nanostructure and their utilities that are found in nature. The exterior of the toucan beak that is made up of overlapping nanosized tiles of keratin, the same protein that makes up hair, fingernails, and horn. The interior of the beak is a rigid foam made of a network of nanosized bony fibres connected by membranes. This allows the beak to absorb high-energy impacts. (<http://pubs.acs.org/cen/news/83/i50/8350toucan.html>). Beetles and flies also have nanostructures that help them stick to walls, ceilings and what appear to be smooth surfaces. 500,000 hairs per toe. Hundreds of nanoprojections (spatulae) per hair. Adhesive force in one foot = 100 newtons.

Then, there have been some extremely revolutionary inventions in the past decade or so, that we need to be acquainted with and much of the research work is still being carried off. Some of these researches includes, Nanocomposites used in making paints, such paints are scratch-resistant, light-weight, and rust-proof, and generates improvements in strength and reductions in weight, which lead to fuel savings and increased longevity. And in 2001, Toyota started using nanocomposites in a bumper that makes it 60% lighter and twice as resistant to denting and scratching Stain-repellent Eddie Bauer Nano-are™, with surface fibers of 10 to 100 nanometers, uses a process that coats each fiber of fabric with "nano-whiskers." Developed by Nano-Tex. Then, there are, sunscreens are utilizing nano particles that are extremely effective at absorbing UV light, Due to the particle size, they spread more easily, cover better, and save money since you use less. They are transparent, unlike traditional screens which are white. These sunscreens are so successful that by 2001 they had captured 60% of the Australian sunscreen market.

One of the most desirable application of nanotechnology that is being explored is in food packaging. "food", being, one of the most important commodity for survival of living beings. Various methods of nano packaging are contributing towards food protection and extension of its shelf life. As we all know that, food Packaging is meant for the protection of food from harmful substances and extending the shelf life. Key characteristics in nano packaging materials are enhanced external, mechanical and thermal properties, protection from any chemical and microbiological effects, additional safety and functionality. Besides, these, the products should be Inert, Cheap, Lightweight, Easy to dispose or reuse, Impervious to environmental storage and transport conditions, resistant to physical abuse. Few examples of such inventions include, Waxy coatings used in fruits, vegetables, meat, to increase shelf life, Sonotec, U.S., has developed an anti bacterial coating for such foods, Nanocrystals embedded



in plastic beer bottles creates a molecular barrier that helps preventing oxygen to escape from the bottles giving the brew a 6 month shelf life. Nanosensor packaging done to incorporate RFID (radio frequency identification) tags for anti-counterfeiting processes. Nansulates used to coat dairy processing tanks and pipes to protect them from corrossion and insulate against heat loss. Use of carbon nanotubes as a sensor to detect toxic substances and spoilage of food, is developed by Georgia tech, U.S. Developing bioswitch for "release on command" preservative packaging. This is intelligent packaging that will release preservative if the food begins to spoil. Silver zeolites used to create anti bacterial polymer composites that offer added stability and slow release of silver ion into stored foods sustains the antimicrobial activity. Chitosan films loaded with AGNPs have better tensile and gas barrier properties.

Nanotech will involve Interlocking ideas, applied knowledge and mechanisms from different fields to fill in the knowledge and data gaps. besides this information I also discussed how these novel nanotechnological advancements could favor ocular (eye) researches and be beneficial for ocular diseases such as amoebic keratitis, that is also one of my research area. countries pioneering in nanotechnological researches, with China pioneering most of the utility based research projects.

Delivering such a talk was a great experience, with students sharing their keen enthusiasm and putting up queries to me. Would like to thank the academic staff of Christ University for making the arrangements. Wish to deliver my next talk again, seeing the interest of the students and university staff, whenever I, next visit India and find time to share some more information, in a more interactive way .

Besides delivering my own talk, I also got the opportunity to attend two, extremely informative lectures at Hong Kong. To meet two such distinguished scientists named Dr. Anella Seddon and other a named, Dr. Jamie Gallengar.

Dr. Anella Seddon, is presently working with the University of Bristol and I got to meet her through British council, who organized her lecture at Hong Kong, at their office. It was a great opportunity to attend her lecture, meet her in person and discuss about nanotechnology and its upcoming developments. Though her interface with the audience was very interactive, I immensely liked the powerpoint preparation of her work and the information displayed. She not only explained the phenomenon in a simple language, but also cited some practical examples as well experiments involving the audience. that served to be an easy way of learning as well as memorizing. She also discussed about the new products that are reaching the market these days and how are they beneficial. This included a whole new range of cosmetics, sunscreens, sport related items, such as tennis rackets, shirts. etc. She also focused her talk on long lived problem of water purification and making it fit for drinking at a large scale. How graphene will act like a miracle material, I came to know through





this lecture only. She also pointed some energy related needs of today's world and provided an answer to them, for example, she told that if we could gather/harvest all the sunlight reaching the earth for one hour, we will be able to provide earth with one full year of energy requirement, so nanotechnology will be helpful in meeting energy demands, but, we still don't know how to do it!! but we may certainly work in the direction to find answers to these questions.

She also touched the areas of drug delivery, where she told us a way how nanobox containing a chemotherapy drug inside will be working only on the cancerous cells leaving the healthy ones, undamaged. these nanoboxes will be "locked" from outside and will be only recognized by cancerous cells.

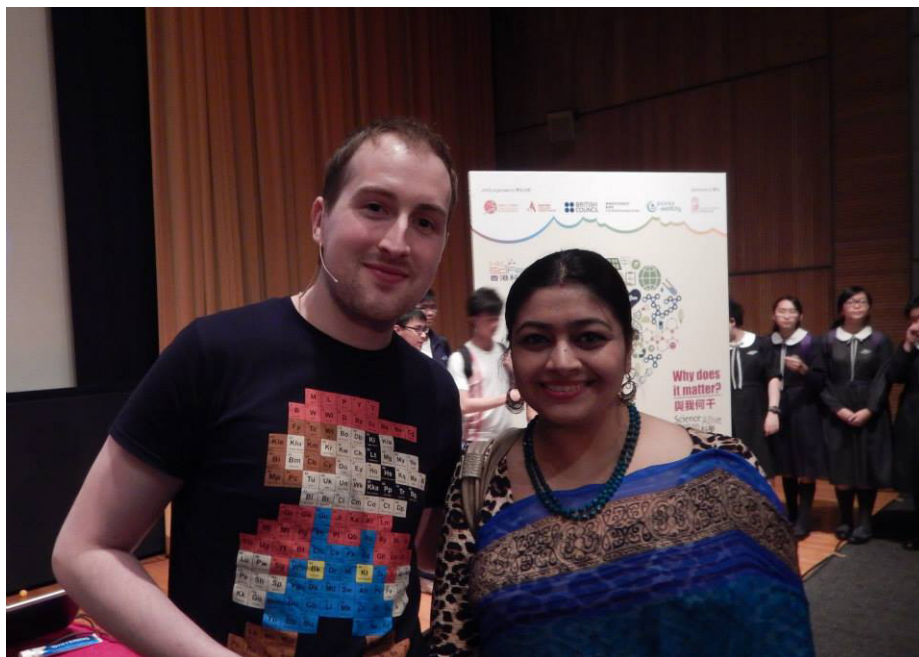
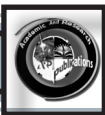
She also discussed how Stanford university is developing self healing skin. After discussing these many ideas, I asked her couple of questions regarding water purification, with special reference to ballast water purification. To which she replied how researchers are being carried out these days to combat such a problem.

The other such interactive lecture was of Dr. Jamie Gallenger. He is a multi-talented person. He is a science communicator, a scientist, a dancer and also works as an Public engagement officer for university of Glasgow. He is an enthusiastic person and very happy to meet new people. He did his PhD. in nano, thermoelectronics. in his own words "*they are clever little devices; they can take waste heat and convert it directly to useful electricity imagine, just by walking around the waste heat, that could be harvested and used to electrical devices, that we carry ! I try and make them work a little bit better than they already do.*"

It was an academically gratifying experience, meeting these two wonderful personalities, both of them working in the areas of nanotechnology. I found it to be worthy of sharing with the readers of this journal and the editor. Such kind of talks not only upgrade our knowledge but also open up our brain's windows, through which we acquire and learn and discover new pathways that lead to new discoveries. I also found that how the scientist are working in the areas of food, environmental science, biological science and medicine, incorporating nanotechnology and trying to find solutions to the problems. We have a good and promising scope for nano science research in present time as well as future. I look forward to meeting these people again and also to attend more of such informative talks and also delivering more talks, exchanging ideas in various ways  
thanking you,

dated: 19<sup>th</sup> May, 2015

Yours sincerely,  
Dr. Kavyanjali Shukla



Dr. Kavyanjali SHUKLA with Dr. Jamie Gallenger at HK science museum



Dr. Anella Seddon, University of Bristol, delivering her lecture at the British council, Hong Kong.



Dr. Kavyanjali Shukla with some of the student listeners



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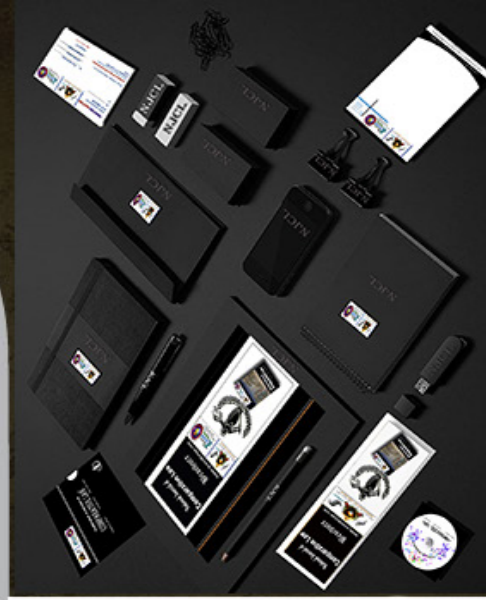


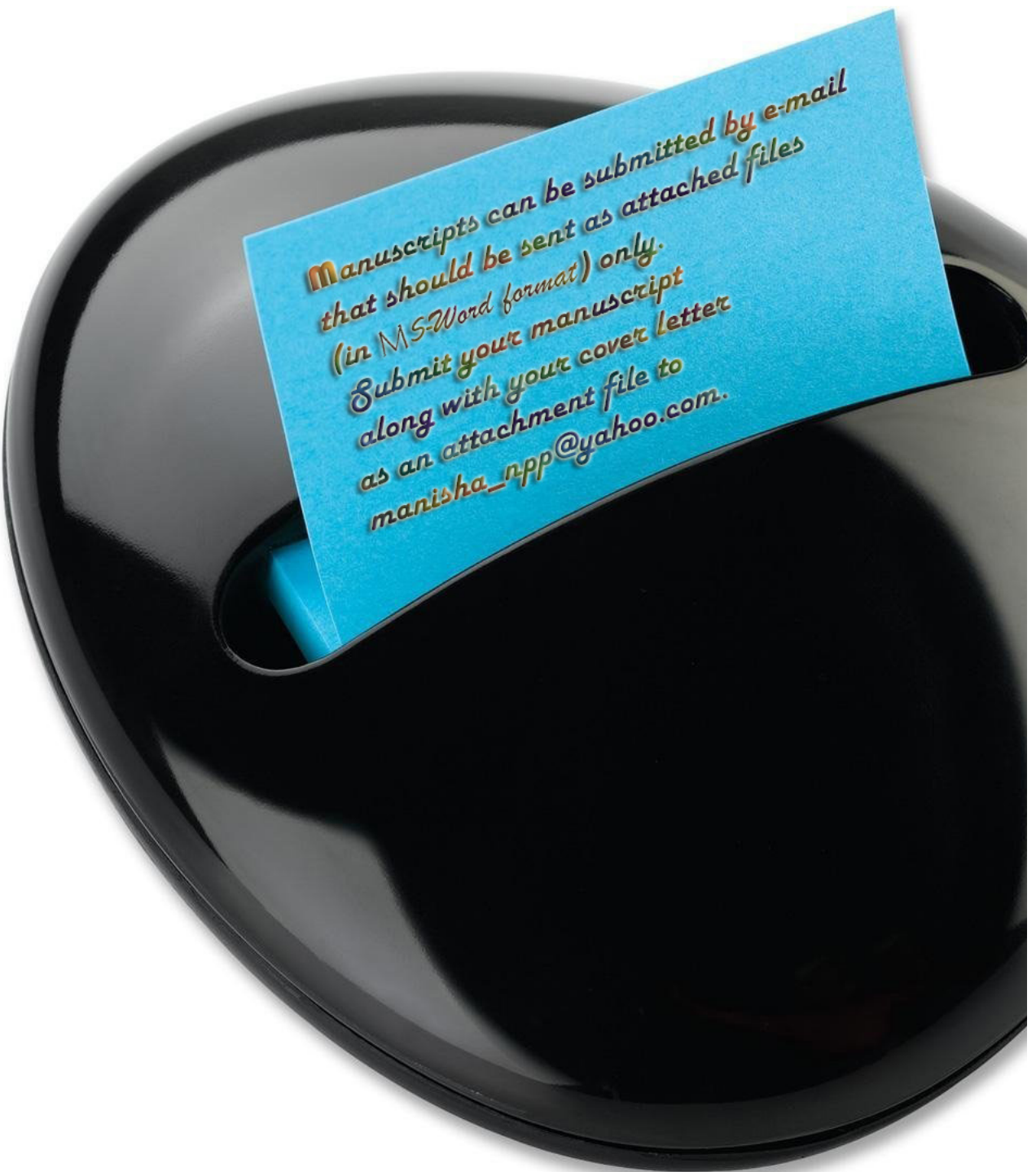
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**From website:**-National Oceans and Atmospheric Administration (NOAA). 1995. Regional Perspectives: Indian Ocean. [www.ncdc.noaa.gov/paleo/outreach/coral/sor/sor\\_indian.html](http://www.ncdc.noaa.gov/paleo/outreach/coral/sor/sor_indian.html), accessed on July 13, 2008.

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